SPECIFIC SURFACE OF MINERAL POWDERS
BY
THE AIR PERMEABILITY METHOD

By
D.E. Pickett

UNIVERSITY OF ALBERTA

FACULTY OF ARTS AND SCIENCE

The undersigned hereby certify that they have read and recommend to the Committee on Graduate Studies for acceptance, a dissertation on "Specific Surface of Mineral Powders by the Air Permeability Method", submitted by Daniel Eugene Pickett, B.Sc., in partial fulfilment of the requirements for the degree of Master of Science.

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Daniel Eugene Pickett

Department of Mining and Metallurgy

University of Alberta

A THESIS

Submitted to the University of Alberta
in partial fulfilment of the requirements
for the degree of Master of Science.

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PREFACE AND ACKNOWLEDGEMENTS

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CONTENTS

Preface

Acknowledgements

INTRODUCTION 1

THEORETICAL CONSIDERATIONS 3

Methods Used for the Determination of Surface 3

Indirect Methods 4
Microscopic Size Analysis
Screen Size Analysis
Sedimentation Size Analysis
Elutriation Size Analysis
Calculation of Specific Surface from Size Analysis
Summary

Direct Methods 26
Gas Adsorption
Chemical Activity
Turbidimetry
Coercimetry
Permeability
Other Methods
Summary

Summary 34

The Air Permeability Method 37

Apparatus 37

Theory of Air Permeability 47

Accuracy of the Air Permeability Method 59

EXPERIMENTAL INVESTIGATIONS 61

Calibration of the Air Permeability Apparatus 61

Preparation of Surface Standards 61

Preparation of Microscopic Glass Spheres 62
Sizing of Spheres by Elutriation 72
Microscopic Evaluation of Specific Surface 95
Discussion of Results 109

The Air Permeability Apparatus 111

Design Considerations
Apparatus
Procedure
Tests on Reproducibility
Calculations of Experimental Error

Calibration Curves for the Apparatus 127

Curves for Glass Spheres
Extension of Porosity Range with Quartz Powders
Extrapolation of Curves for High Surface Values
Tests on Composite Samples

Accuracy of the Calibrated Apparatus 143

Accuracy of the Calibration Curves
Error in Permeability Tests

Size Surface Measurement on Pure Minerals 146

Size Surface Measurements on Quartz Powders 146

Procedure
Tests and Results
Discussion of Results

Standard Procedure for Pure Minerals 154

Standard Elutriation
Surface Determination

Tests on Pure Minerals 157

Quartz
Ground Pyrex Glass
Pyrite
Galena
Cassiterite
Discussion of Results

Application to a Heterogeneous Powder 167

Surface Measurement
Size-Surface Measurements
Discussion

Summary 170

CONCLUSIONS 171

RECOMMENDATIONS 174

Appendices

References

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The object of this investigation is to develop a simple and easy application of the permeability method of surface determination which can be used in the practical evaluation of efficiency of mineral dressing operations.

In mineral dressing, extractive metallurgy, and other processes of the mineral industry, the preparation and treatment of vast quantities of powdered material is necessary. In all cases the efficiency of these processes seems to be related in some way to the amount of surface in the material treated. For example:- in grinding, the energy consumed is a direct function of new surface created; in flotation, reagent consumption is a function of the surface of the mineral particles; in leaching, the rate of dissolution of mineral varies directly with the surface exposed to the chemical; and in flash roasting, the completeness of oxidation is a function of the surface. Therefore the development of a method to measure the surface area of powders is certain to be of importance in the treatment and utilization of powdered materials.

Many investigators have attempted to set up mathematical formulae for the evaluation of surface in ground products, but have been more or less unsuccessful in their efforts, because in such a mathematical treatment the necessary fundamental theory relating surface to particle shape and size is lacking. Although theoretical assumptions as to shape and size can be made, the accuracy obtained

is not good and does not seem to warrant the tedious mathematics involved. The possibility of extending fundamental knowledge, so that a well founded mathematical treatment of surface determination can be evolved, should not be overlooked, but until such data are available, some other method is needed to serve the mineral industry.

For the moment it would seem that a method is needed which is simple in application and eliminates theoretical assumptions and statistical approximations. In this investigation a start has been made to evolve such a method. It involves the use of the permeability method of direct surface determination and circumvents theoretical considerations of shape and size by correlating all data to a common standard of unquestioned validity.

THEORETICAL CONSIDERATIONS

In order to assess the merits of the many methods which have been used for surface determination, a study of relevent literature was made. The results of this study indicate that for the purpose of routine testing the method with the most favorable characteristics is the air permeability method.

A further detailed study of the theory and application of the air permeability method was made to determine the controlling principles and possible range of application.

In this section accounts of these studies are given.

METHODS USED IN DETERMINATION OF SURFACE

A discussion of the various methods of surface determination is given here to indicate the limits of application of these methods, and to show the advantages of the permeability method over the other methods.

The methods, in general, utilize all the physical properties of particulate matter and attempt to relate these properties to surface. Their accuracy depends on two factors:

(1) The measurement of the physical property.

(2) The relationship between the property measured and surface. Inaccuracy in either factor may cause error in the resulting surface value.

The methods may be classified generally into two groups:

1. Indirect Methods.
2. Direct Methods.

INDIRECT METHODS

Indirect methods of surface determination involve the measurement of the size of the particles and subsequent mathematical calculation of the surface.

Normally the calculation is carried out from some linear dimension of the particles. Any method therefore requires the following information:

(a) The linear dimensions of the particles.

(b) The distribution of the sizes by weight.

(c) A factor relating linear dimension to surface.

The common indirect methods for obtaining the required data are:

1. Microscopic methods.
2. Screening methods.
3. Sedimentation methods.
4. Elutriation methods.

Since any indirect determination depends largely upon the final method of calculation, this phase will be considered after discussing the individual methods of size analysis.

Microscopic Size Analysis

The most commonly used method of obtaining the particle size distribution of a powder is by microscopic measurement and count. G.L. Fairs of Imperial Chemical Industries[(1)] states that the microscope affords the only reliable method available for size analysis in the -5.0 micron to +0.3 micron range.

Figures in parenthesis refer to bibliography at end of this thesis, e.g. (5-118) indicates reference number 5, page 118.

Details of technique are more or less standardized and the American Society for Testing Materials Specification E20-33T[2] gives the general procedure. Specific methods are given by Heywood[3], Stienherz[4], Taggart[5-118], Krumbein[6], Pieters and Hovers[7], British Coal Operators' Research Association[1], and others. The chief factors to be considered are:

(1) Accurate sampling.

(2) Complete dispersion.

(3) Attainment of uniform field.

(4) Adequate magnification and definition of particle boundary.

(5) Accurate method of measurement.

(6) Proper selection of grade limits.

Sampling, in addition to all the normal difficulties discussed by Taggart[5-1], is made more difficult by the small size of sample required. Microsplitters of various designs are used but the error in sampling can be great.

Dispersion is a matter of proper choice of mounting medium and good dispersion technique. The medium should be fluid enough to permit of mechanical dispersion and still viscous enough to suspend particles after dispersion until transferred to the slide. Liquids which possess these qualities are glycerol and water, glycerol and alcohol, glycerol jelly, glycol, balsam in xylol, rubber cement, styrax in xylol, saponin, etc. If a permanent fixed mount is required by the method, the diluent for the medium should be volatile. Mechanical dispersion in the medium must not abrade the material but must result in complete separation of individual particles with no flocculation. Several

techniques are used, stirring with a high speed agitator or brush being recommended.

Uniformity of field is dependent upon the medium and the mounting technique. Several techniques are used, all subject to criticism by contemporary investigators, but the final test of a good technique is the uniformity observed under the microscope. Fine powders between close limits of size are most easily handled. Extremely heterodisperse powders containing coarse material are very difficult to handle.

Particle definition depends upon the microscope used and the difference in refractive index between the medium and the material. It has been found by Fairs(1) that the lower limit of the microscope is 0.3 microns, and for sizes below 1.0 micron, monochromatic light and a very good condenser must be used to obtain proper definition. For powders finer than 0.3 microns, Arnell(8) claims accurate results with the electron microscope.

Magnification and method of measurement go together. The A.S.T.M. Specification prescribes that measurement of particles below 1.0 microns must be accurate to 0.1 microns, so magnification must permit of this. The filar micrometer ocular, the Whipple disc, or the Patterson graticule, are all used and recommended for direct measurement under the microscope. This is an extremely fatiguing procedure and it has been found by Imperial Chemical Industries(1) that two workers are required at each microscope for satisfactory work. Even then the time required is excessive since 200 to 300 particles must be counted and measured. A more satisfactory method, as regards time and accuracy, is to prepare photomicrographs with a magnification which will permit of measurement

with a scale. This requires laboratory facilities not always available. Projection of the field of view upon a screen is also used[3].

Choice of method is determined by the apparatus available, the nature of the material, and the accuracy required. Method of calculation also determines the data required and the method to be used.

The selection of grade limits affects the accuracy of the resulting size distribution, and determines the method of calculation.

Although considerable error is inherent in the microscopic method, from a practical viewpoint the method is sufficiently accurate for the purpose, since conversion of the data to surface involves an assumed shape factor which tends to destroy any accuracy obtained by more tedious microscopic procedure. The method will not give true values except for powders of spherical or other regularly shaped particles.

Screen Size Analysis

Screening is the most convenient method for determining particle size distribution of plus 200 mesh material. The screens and the technique to be used are standardized by the American Society for Testing Materials[9], the U.S. Bureau of Standards[10], and the British Engineering Standards Association. The specifications for screens for the three standards are given by Taggart[5-103].

Although a system for calculation of surface from screen data was developed by Coghill[11-261], the determination is of little use since the surface of the coarse screen sizes is of very little importance in ore dressing when compared to the surface of the subscreen material.

Sedimentation Size Analysis

Sedimentation methods of sizing are based upon the principle that different sized particles of the same specific density and shape

have different settling velocities in a fluid. This results in a more rapid settling of coarser particles to the bottom of the sedimentation chamber. Successive samples of the suspension taken at a given level will measure this velocity and a size distribution curve can be calculated. Theoretically the method should be very accurate but practically several factors make it approximate. These are:

(1) Limited accuracy of formulae

(2) Shape factors

(3) Temperature and specific density variations

(4) Difficulties of measurement.

Settling Laws: The mathematical laws which have been derived to calculate the settling velocity of a particle of given size differ considerably for various size ranges. The most generally accepted is Stokes' Law which is derived from theoretical considerations involving the coefficient of resistance to movement of a particle in a fluid, and the Reynolds number relating movement to viscosity and density of medium. In its usual form Stokes' Law expressed mathematically for spheres is

$$v = \frac{\frac{2}{9}(d_1 - d_2)\, gr^2}{U}$$

where v = velocity of settling

d_1 = density of material

d_2 = density of fluid

U = viscosity of fluid

r = radius of sphere

g = gravitational constant

This law applies only over a restricted range where the relative motion of liquid past the particle is streamline flow, and

holds for spheres between 85 microns and 0.1 microns in water.

For particles below 0.1 microns, the conditions and equations for streamline flow do not hold since the particles are small enough to slip between the molecules of fluid. Therefore the terminal velocity increases as given by Cunningham's correction to Stokes' Law[11-17].

For particles settling under conditions of turbulent flow the Newton Law is postulated:

$$v = K_t\sqrt{\frac{d_1 - d_2}{d_2}.D}$$

where K_t is a factor varying with the shape of the particle and equal to 25.4 for spheres. For other shapes, values of K_t are tabulated by Dallavalle[11-16].

For conditions of settling between streamline and turbulent flow no equations are given which seem wholly satisfactory. A common assumption used by Oseen, Goldstein, Budryk, and Waddell[12-172], is that the resistance to fall is approximately equal to the sum of both turbulent and streamline resistances. The individual formulae do not fit experimental data unless an empirical coefficient is introduced.

Formulae covering the whole range of particle velocities have been used by Allen[11-16], Needham and Hill[13], and Gaudin[12-175]. These formulae give the general relationship between Reynolds Number, R_e, and the Resistance coefficient, C_r. In practice the value of C_r is obtained by calculation, from tables, or from graphs, and substituted in a general formula for settling velocity.

Other expressions for settling velocity and more detailed mathematical treatments are given by Dallavalle[11-Ch.II], Krumbein and Pettijohn[6-VI] and Gaudin[12-VIII].

Shape Factors: Particle shape affects the results of the above equations considerably. Values to be used for spheres and other mineral shapes under streamline and turbulent conditions are tabulated by Dallavalle(11-16). Richards gives equal settling ratios by which the velocities of fall of typical mineral particles are referred to spheres. These values are tabulated by Gaudin(12-187), and Taggart(14).

Conditions of Test: To obtain consistent results in sedimentation analysis certain conditions must be maintained and standard procedures must be followed. The standard conditions necessary are:

(1) Stream line flow.

(2) Free settling.

(3) Freedom from convection currents.

(4) Perfect initial dispersion.

(5) Maintenance of dispersion.

(6) Freedom from disturbance of conditions during test.

Streamline flow conditions have been found best for sedimentation work since the settling laws fit these conditions most closely. These conditions are obtained by using fluids related to particle size so that the Reynolds number is kept below 4.0(13). For particles with diameters between 0.1 and 85 microns water is satisfactory. Air is suitable for very fine particles and has been used by Pieters and Hovers(15).

Free settling without hindrance from walls or other particles is necessary. Therefore the sedimentation chamber must be large enough to minimize wall drag. Krumbein(6) recommends that the tube diameter be not less than 5 centimeters, and that the density of the suspended solid

be not more than 25 grams per liter to ensure free settling conditions. Andreason recommends a 3 to 5 per cent. suspension by weight(5-123). The A.S.T.M. standard method for soil testing(16) specifies a 3 per cent. suspension.

Convection currents can cause a very serious error particularly in the small size fractions. To control temperature and convection currents the sedimentation chamber is normally enclosed in a thermostat, if minus 10 micron material is under test.

Perfect initial dispersion of powder in the medium is assumed as a condition for most methods. Mechanical dispersion of some type is necessary to break up flocculated material, but should not produce fines. Common methods are agitation in a bottle, or in some cases in the apparatus, or stirring with a high speed stirrer. The agitation must be continued until results indicate no flocculation of material. A chemical wetting agent added to the material assists dispersion. Methods of determining the degree of dispersion attained, are discussed in the report of the British Coal Research Associations Conference on Particle Sizing(1).

Perfect dispersion must be maintained throughout the test or the results will be incorrect. Low density of suspension assists in this. A dispersion agent is usually used. Common dispersion agents are listed by Taggart(5-117). Dispersion agents of different types are discussed by W. Fryer(1), with special emphasis on details of concentration and time of contact. Pieters and Hovers(7) tabulate results of tests on various dispersing agents at different concentrations.

The rate of sedimentation should not be disturbed when the reading is taken since time is cumulative in most methods. If the

procedure necessitates the removal of a portion of the suspension at a specified time, or the introduction of an instrument, this must be done very carefully so that sedimentation conditions are not disturbed. The introduction of an instrument which reduces the cross section of the chamber may cause considerable wall effect.

In addition to ensuring the above conditions certain techniques are necessary to maintain accuracy. Sampling must be representative and accurate since samples are small for most methods. Accurate determination of the specific gravity of the powder is very difficult and requires very accurate apparatus and careful technique.

Sedimentation Methods: Several methods have been devised for sedimentation analysis which have a limited accuracy depending on the fulfillment of the above conditions, and upon the accuracy of the specific methods used to measure sedimentation rate. These methods are classified by Schweyer and Work[17] as being either (a) Increment, or (b) Cumulative Methods.

(a) Increment Methods. In the Increment methods the change in concentration at a given level is measured. The assumption is made that this change in concentration is caused by depletion of particles whose velocity is such that they have fallen below the point of measurement in the measured time interval. If the settling depth in the time 't' seconds is 'H' centimeters, then the velocity of such particles must equal or exceed H/t. Therefore substituting in Stokes' Equation the limiting diameter of such particles is given by the formula

$$D = \sqrt{\frac{1,837,000\ U.}{d_1 - d_2} \frac{H}{t}}$$

where D = diam. of particle in microns

U = viscosity of fluid in poises

H = depth of measurement in centimeters

d_1 = Particle density, grams per c.c.

d_2 = Medium density, grams per c.c.

t = time of settling in seconds.

This equation holds for spheres[18].

The actual measurement of change of concentration may be done by several methods:

(1) Pipette methods.

(2) Hydrometer methods.

(3) Pressure methods.

(4) Photometric methods.

(5) Photographic methods.

(6) Centrifugal methods.

A full discussion of these sizing methods may be found in any text book of sedimentation and sizing analysis.

(b) Cumulative Methods. In the cumulative methods the same assumption is made as to size of particle as in the increment method. However the quantity of material accumulating at a certain level is measured, rather than the loss of material at a certain level.

Measurement of material accumulation is by:

(1) Sedimentation balance.

(2) Pressure manometer.

These methods are subject to considerable error since uniform conditions cannot be maintained throughout the chamber.

Elutriation Size Analysis

Although based on the same laws of particle settling as sedimentation methods, and sometimes considered a sedimentation method, elutriation differs in that, in addition to a size analysis, separate sized fractions are obtained as products of the operation. Where only a size analysis is required, sedimentation is more accurate and much simpler. If subdivision of the powder into sized fractions is required, elutriation is necessary.

Sized fractions are desirable for surface determination since it has been proven that different size fractions have different sphericity factors. Accurate microscopic measurement of powders requires that the powder be first divided into fractions of limited range, so that good slides can be prepared and all particles will be in focus. Also if distribution of surface in a powder is to be determined by a direct method, the powder must be divided into fractions(2)(24).

The same assumptions as to particle settling velocity are made as in sedimentation. The same general conditions as listed on page 10 are necessary.

Elutriation methods may be divided into three classes:

1. Fractional settling.
2. Rising current elutriation.
3. Dynamic air elutriation.

Fractional Settling: In this method normal sedimentation is allowed for a predetermined time until all particles coarser than the required size have settled to the bottom. Then the suspension medium with the suspended fine material is siphoned or decanted off. Since considerable

fine material settles out with the coarse material, the settled portion must be repulped in fresh medium and the operation repeated until the required clean coarse fraction is obtained.

The accumulated suspension, now containing all material below the size required in the first stage, is next repulped, and the next finer fraction obtained by use of a greater settling interval. This cycle is repeated using a longer settling time for each cycle until the required number of fractions is obtained. To obtain greater accuracy each fraction may be retreated to remove any coarse particles accidentally carried over in siphoning or decantation.

The method is capable of very good sizing and is the only method by which a highly accurate fractionation can be carried out. Battelle Institute[19] sizes microscopic glass spheres by this method, preparing sized fractions 90 per cent. within a 4 micron range. Stienherz[4] prepared fractions for microscopic study by this method using a train of jars with vacuum siphoning arranged to carry out several operations simultaneously, thus reducing the time and volume of fluid required. But by any arrangement the method is very tedious and is only to be recommended for analytical work where high accuracy is required.

Rising Current Elutriation: This is the most common method for sizing a powder into several fractions. The fluid used may be either liquid or air. A rising current is produced in the elutriation chamber with a velocity equal to the terminal settling velocity of the smallest particle desired to remain in the chamber. All the smaller particles of lower terminal velocity are carried over in the overflow.

For calculation of this particle size some settling law must be assumed. Normally Stokes' law is used assuming stream line flow. Theoretically then, the accuracy of the sizing will depend upon the time allowed for particles, only slightly smaller than the limiting size, to rise from the bottom of the chamber to the overflow level. Actually several factors decrease the accuracy considerably.

(a) Liquid Current Method: It is practically impossible to produce absolutely streamline flow in a chamber without using liquids of high viscosity. The entrance velocity must be dispersed uniformly across the chamber and then must be kept uniform. If the first condition were obtained the drag of layers of liquid against the wall would destroy the uniformity. Also as the liquid approaches the overflow level, induced currents due to outlet velocity destroy the streamline effect. The result is verging upon turbulent flow, as proven by Needham and Hill[13] who found the calculated balancing velocity in elutriation was not equivalent to the particle terminal velocity, with Reynolds numbers in the range approaching those found in turbulent pipe flow. Streamline flow would necessitate a plane velocity front. Evidence that the velocity front in straight tube elutriators approaches parabolic is given by Taggart[5-117]. However, as pointed out by Taggart, lack of stream line flow is not a serious cause of inaccuracy except that it causes some overlap of fractions and makes the theoretical determination of limiting size approximate. The actual limiting size can be determined by microscope. At a given velocity results are reproducible.

Assuming a satisfactory accuracy of cut between overflow and retained material, an increase in current velocity will produce another

fraction of coarser material. The overflow may be elutriated at successively lower velocities to give finer fractions. In this way any number of fractions can be produced, and weighed to obtain a sizing analysis. With a uniform fluid velocity, a series of tubes of increasing diameters will produce a series of fractions decreasing in limiting size in one operation. This latter arrangement is called a series or multiple-tube elutriator.

The same disadvantages as to time and volume of fluid affect the utility of elutriation although not as seriously as in the fractional settling method. Time for fine fractions may be as great as 24 hours with some types of apparatus. This may be reduced by shortening the actual sizing column. Liquid volume is greater with coarse sizes and less with finer sizes.

Design of liquid elutriation apparatus has undergone many changes in attempts to overcome overlap of fractions, reduce the time and liquid volume required, and increase the size of sample. The various designs are illustrated and discussed very thoroughly by Krumbein and Pettijohn[6-VI], and by Taggart[5-109]. Tube designs vary from the long cylindrical tube used by Gross[20] and Probert[21], to special glass shapes developed by Andreason[6-VI]. The Andrews Kinetic Elutriator[5-112], and the U.S.B.M. Elutriator[5-113], employ complicated mechanical apparatus which is expensive and is only slightly more accurate than the simpler types. Technical modifications for stirring, baffling, and measuring current velocity are numerous. Multi-tube elutriators of the Mount Morgan type[5-112], Andreason type, and the Dutch States Mines type[7] are normally used for routine testing.

(b) Air Current Method: Air elutriation has the advantage of greatly reducing the time required for analysis since the terminal velocity of falling particles in air is much greater than in liquid. Loss of fractions in filtering and drying after elutriation, a common cause of error in wet elutriation, is avoided in air elutriation. The method is therefore better adapted to routine testing.

There are three main disadvantages which make this method less accurate than liquid elutriation:

(1) Fluid movement is definitely turbulent flow.

(2) Flocculation of fines is common since no dispersing agent can be kept in solution.

(3) It is almost impossible to prevent clinging of fines to the sides of the tube due to electrostatic effect.

These disadvantages cause greater overlap of fractions than in the liquid method.

The same principles of design apply as for liquid elutriation. The common type of single tube air elutriator is that of the U.S. Bureau of Standards described by Taggart[5-113]. A similar type with simplified flow measurement apparatus is used by the Dutch States Mines[21]. Dallavalle[11-72] uses a tube with centrifugal dispersion by compressed air jets and a cyclone type collector.

The most successful multi-tube type is the Haultain Infrasizer employing conical tubes and ball-jet air inlets. This apparatus is used for rapid routine testing by many ore dressing laboratories. The high speed jet breaks up flocs. The apparatus is described by Taggart[5-116] and operating characteristics are discussed in a report by Lake Shore staff[22].

Dynamic Air Elutriation: The Roller air-elutriator[23] operates on a dynamic principle by which an air jet in a U-tube sorts the material into two fractions, the finer being carried over into a collector. Elutriation producing one fraction takes up to six hours but the accuracy obtained is considerable better than that obtained with rising current types.

Comparison of the relative accuracy of the various types of liquid and air elutriators is difficult since there are no standards by which the absolute size of the product can be measured. All types have disadvantages which make them unsatisfactory with certain types of material. In all air elutriators clinging fines and flocculation make determinations of minus 10 micron material useless. Also control of air flow is very difficult without intricate control apparatus.

Calculation of Specific Surface from Size Analysis

In discussions of surface of ground products, the unit of surface is the square centimeter, and the amount of surface in one gram weight of material is called the Specific Surface. This terminology is now universally accepted as standard although other meanings for 'Specific Surface' may be found in earlier technical writing.

The calculation of specific surface of a powder is not a simple problem due to shape considerations, and will therefore be developed here by discussion of the cases which may be encountered in order of increasing difficulty. These cases are:

1. Powders composed of spherical particles.
2. Powders composed of particles of known shape.
3. Powders composed of irregularly shaped particles.

Spherical Particles: The simplest case is that of a powder composed of uniformly sized spheres of one material. If the material has a specific density 'd' and the spheres are all of diameter 'D' microns, then the specific surface 'S' in square centimeters per gram will be accurately equal to the surface area of one sphere times the number of spheres in one gram, or

$$S = \frac{\pi D^2}{(10^4)^2} \times \frac{(10^4)^3}{\frac{1}{6}\pi D^3 d}$$

$$= \frac{60000}{dD} \qquad (1)$$

Consideration of the second case where the spheres are not all of the same size, involves either the establishment of a mean diameter or the summation of the areas of each size grade or fraction. Two problems affect the accuracy: the size limits of each grade, and the mathematical approximation in computing the mean diameter of each grade or the mean diameter of the whole sample.

Any systematic subdivision into grades may be used but the most commonly accepted method is to use grades in which the limiting sizes of successive grades bear a constant mathematical relationship. Theoretically the mean size of each grade should be weighted toward the surface mean but this involves tedious calculation and the normal method is to use the arithmetical average of the grade limits as the mean, keeping the limits of the grade as close as practicable.

If then the sample is divided into grades of mean diameters D_1, D_2, D_3, etc., microns, and the number of particles in each grade are n_1, n_2, n_3, etc., the total surface in each grade will be,

$$\frac{\pi}{10^8} n_1 D_1^2, \quad \frac{\pi}{10^8} n_2 D_2^2, \text{ etc.}$$

The total surface of the sample will be,

$$\frac{\pi}{10^8} \sum n\, D^2 \quad .$$

Similarly the total weight of the particles will be

$$\frac{1}{6}\, \frac{\pi d}{10^{12}} \sum n\, D^3 \quad .$$

The specific surface for the sample, with small error, will be equal to the total surface divided by the total weight, or

$$S = \frac{60000}{d} \times \frac{\sum n\, D^2}{\sum n\, D^3} \quad .$$

Comparing *with* equation (1) it is evident that $\dfrac{\sum n\, D^3}{\sum n\, D^2}$ is equivalent to the mean surface diameter, D_s, which may be found directly from the distribution data(5-143), so that for unsized spheres of common specific density

$$S = \frac{60000}{d\, D_s} \quad , \qquad (2)$$

$$\text{where } D_s = \frac{\sum n\, D^3}{\sum n\, D^2} \quad . \qquad (3)$$

The accuracy of this value depends on the approximation involved in the mean diameter of the individual grades.

Particles of Known Shape: Since the derivation of equation (2) depends upon the particles being spheres it cannot be used in the case of powders of nonspherical particles without consideration of the effect of shape. In the case of spheres the factors relating surface and diameter are known. Similarly for cubic particles there is a fixed relation between surface and diameter. But for any other regular geometric shape the concept of diameter is not rigidly defined. For such regular geometric shapes, separate formulae for specific

surface can be derived based on the linear characteristics of the particle.

As this method, if applied to all possible shapes, would involve a great many equations, several attempts have been made to relate some characteristic dimension of the particle to the surface of a sphere so that equation (2) can be used. Then if some equivalent spherical diameter, D_e, can be derived,

$$S = \frac{60000}{d\ D_e} . \qquad (4)$$

This necessitates a factor for each shape relating the dimensions of the particle to D_e. For example, for cubes the equivalent spherical diameter will be slightly greater than the cubical diameter D_c as shown by calculation. Since D_e must be the diameter of a sphere with equal surface to the cube,

$$\pi D_e^2 = 6\ D_c^2$$

$$\text{or } \frac{D_e}{D_c} = \sqrt{\frac{6}{\pi}} = 1.382$$

$$\text{or } D_e = 1.382\ D_c .$$

Then for a powder composed of cubic particles,

$$S = \frac{60000}{d\ D_e} = \frac{60000}{d(1.382\ D_c)} ,$$

where D_c equals the mean surface diameter for a heterodisperse powder.

Similar calculation is possible for all shapes if D_e can be related to a certain dimension by a factor.

However since diameter is an artificial term for regular geometrical shapes other than spheres or cubes, a more rational system has been evolved relating surface to volume. In this system the ratio of the surface of a sphere of equivalent volume to the surface of the

actual particle is defined as the sphericity. Values of sphericity for common geometric figures are tabulated by Taggart[5-141] and Gaudin[12-195] and vary from a maximum of 1.0 for spheres. To relate this concept to calculation of specific surface from linear measurements requires the calculation of the equivalent volume of the particle.

Irregular Particles: From the consideration of theoretical powders composed of regular geometric shapes, some idea can be obtained of the difficulty of obtaining an accurate value for specific surface when the shape is known. When an actual powder of one material composed of natural irregular shapes is considered, the number of assumptions and approximations can cause considerable error in the specific surface determination. The uncertainty of these calculations is reflected in the multitude of theories, formulae, and methods of determining particle size and surface which are published in the technical literature.

The first stage in obtaining the specific surface of a natural powder is to measure some characteristic of the individual particles which is representative of the mean size of all the particles. This statistical characteristic is normally called the particle diameter.

In microscopic work several "diameters" have been proposed by investigators. None of these takes full account of the thickness of the particle which is something less than the diameter obtained from measurement of the particle as it rests on the slide. The most commonly used measurement is Martin's statistical diameter which is the diameter which bisects the area of the particle measured parallel to a fixed direction. This diameter has been shown by Heywood[3] and Stienherz[4] to differ from the equivalent circular diameter, D_c, by

less than two per cent. The equivalent circular diameter or mean projected diameter is the diameter of a circle with the same area as the projected area of the particle. Another diameter measured is the equivalent spherical diameter, D_e, the diameter of a sphere of equal volume. This requires measurement of the volume of the particle, by counting the number of particles of powder making up a given volume of material. Factors for comparing equivalent spherical diameters are given by Taggart[5-140]. Feret's statistical diameter is also used, which is the number mean of all maximum diameters measured parallel to a given axis. Waddell[5-141] measured the diameter of the sphere circumscribing the particle.

The conversion of the nominal diameter obtained, into a value of specific surface is not as simple as for regular geometric shapes. Several systems have been proposed. Of these the two most commonly used are the two described above for regular geometric shapes involving D_e or D_c.

If the equivalent spherical diameter, D_e, can be determined, equation (4) gives the specific surface. The accuracy of the result depends upon the degree of approximation involved in the equivalent diameter.

Given a statistical diameter, D_c, a factor of sphericity can be used to obtain the surface of the equivalent sphere and the specific surface can be calculated.

Heywood[3] proposes a method in which a factor for particle area and a factor for particle volume are assigned to regular geometric shapes. By judgement under the microscope the typical shape is selected. Martin's diameter is measured and the factors are applied to obtain the specific surface.

To use any of the above methods an approximate shape factor must be determined. Many actual measurements of shape factors are published for specific materials but are only applicable to the material measured. These measurements are all made on macroscopic fragments of crushed material and are no doubt accurate. Table 5 in Taggart[5-141] is typical. However the application of these factors to microscopic material is impossible since shape factor varies with increasing fineness and with the method of powder production.

Derivation of a statistical mean from distribution functions have been proposed by Bond[24], Gaudin[12], Rosin and Rammler[5-145], and Roller[25]. These involve complicated distribution functions which do not agree as to results and are only accurate for theoretical powders with ideal size distributions. For actual powders a sizing analysis is necessary and calculation of D_s is more reliable.

Summary

In general the method of calculation of specific surface from a size analysis must depend upon the method of sizing. The accuracy of any one method may be satisfactory only for the powder to which it was originally applied and may be entirely inadequate for other powders.

Schweyer and Work in discussing the accuracy and use of sizing methods recommend wet screening as the best method down to minus 325 mesh, and sedimentation below this size. For speed and accuracy they consider the Andreason pipette method[5-122] best and this is also the opinion of other investigators[1]. For rapid control work, Schweyer and Work recommend either the Hydrometer method or the Gross Turbidimetry photometric method.

Pieters and Hovers (7) (21) investigated all common methods and concluded their States Mines Pipette method was most accurate, claiming relative accuracy within 1 per cent., compared with microscopic and elutriation analysis of spherical glass powders. However the actual accuracy is in doubt since both microscopic and elutriation methods are subject to errors.

It is evident that, regardless of the method of sizing the particles, the only accurate calculation of specific surface which can be made is that on spherical particles. These lend themselves most readily to sizing by microscope which is the most accurate method of measurement. A spherical powder sized by microscope should therefore be an accurate standard.

DIRECT METHODS

Direct methods of specific surface determination differ from indirect methods as they measure some property directly related to surface without the intermediate determination of size. The inaccuracies of calculation of surface from diameter involving statistical average and shape factor are thus avoided. However the mathematical relationship between the surface property and specific surface is not always any more accurate than the relationship between diameter and specific surface. The physical, chemical, or optical relationships are either deficient in fundamental theory or too complicated for normal use without considerable approximation.

Therefore all direct methods depend upon empirical constants of limited accuracy or limited range of application. Each method has a size range over which it may be considered reasonably accurate, but

no one method covers the whole range from colloidal to macroscopic material. A possible exception to this limit of range is the gas permeability method which has recently been extended down to particles with a volume surface of 4.6×10^5 square centimeters per cubic centimeter, corresponding to an average particle diameter of less than 0.2 microns(24).

The direct methods of surface determination are:

1. Gas adsorption method.
2. Chemical activity methods.
3. Turbidimetry method.
4. Coercimetry method.
5. Permeability methods.
6. Other methods.

Gas Adsorption Method

This direct method of surface determination is discussed in detail by Emmett(25), Gaudin and Bowdish(26), Dallavalle(11-269), and Taggart(5-130).

The quantity measured is the volume of gas adsorbed on the surface when a known volume of gas is admitted to a highly evacuated sample. The apparatus consists of three parts:

(1) A vacuum system capable of high vacuum to remove all gas from the sample surfaces.

(2) A gas burette to measure the original quantity of gas admitted to the sample.

(3) A manometer system to measure the pressure of the residual gas after adsorption.

From the measurements of pressures and volumes, the volume of gas adsorbed on the powder can be calculated with good accuracy. The surface area necessary to adsorb this quantity of gas must then be calculated. Here the accuracy of the method is lost. The assumption that the gas forms a unimolecular layer on the surface cannot be proven. Experimental evidence(27) shows that there is a concentration of adsorbed molecules at edges and corners corresponding to the free surface energy. Therefore although the assumption is probably not much in error for spherical or coarse particles, considerable error will occur with fine powders.

The basis of calculation is the Van der Waal adsorption isotherm which is extrapolated to give a value for a monomolecular layer within 15 per cent. The final surface value can hardly be considered more accurate than this, and considering the other assumptions the result is probably of correct order only.

However in spite of the approximate result the method has some distinct advantages over other surface determination methods. It can be used with the same accuracy for all heterogenous powders. Also it measures all surface exposed, including small cracks and pores missed by other methods. The significance of this is shown by a surface measurement of 301 square centimeters made on a 2 centimeter cube of galena by Yavasca(28). Crushing of the galena eliminated these cracks progressively until at 200 mesh the measured surface was only 3 times the microscopic calculated surface. Similar results were obtained with pyrite. Such measurements have a tremendous effect upon the efficiency of crushing or grinding based on surface measurement.

[illegible]

The basis of calculation [illegible] tion is [illegible] within 15 per cent. The [illegible]

[illegible]

[illegible] distinct advantages over [illegible] with the same accuracy [illegible]

[illegible]

Chemical Activity Methods

Several direct methods of surface determination are based on chemical activity. By the principles of reaction kinetics the rate of a chemical reaction in which a liquid acts upon a solid is proportional to the surface of solid exposed to the liquid. Therefore if at any instant the rate of the chemical reaction can be measured, the surface area exposed at that instant can be calculated. The rate of reaction can only be determined by change of concentration over a period of time. Since the surface exposed is diminishing rapidly with time the initial rate and initial surface can only be found by extrapolation.

Other aspects of reaction kinetics besides surface affect the actual rate. The rates of diffusion to and from the surface must be greater than the rate of reaction. Otherwise the actual concentration of chemical at the solid liquid interface can only be assumed.

The method has been used with success by Martin(29), and Gross and Zimmerley(30), to measure the surface of quartz with hydrofluoric acid as the reacting chemical. The basis of correlation between surface and concentration change is a factor obtained by test on a plane surface of quartz. Close control of initial concentration and technique is necessary.

The surface of carbonates has also been determined by this method using dilute hydrochloric acid, but the gaseous product gives considerable error.

The dissolution method gives a measure of all surface exposed although the diffusion of reagent into cracks and crevices must introduce an error. Also as in adsorption methods an error is introduced at corners and edges since free surface energy affects the rate of reaction.

The chemical method is limited to very few pure minerals and therefore is only a research tool and not adaptable to routine mill testing.

Turbidimetry Method

In this method the quantity measured is the light obscuring power of the powder when suspended in a liquid. The sample in suspension is placed in a chamber and is agitated to ensure uniform distribution. A beam of parallel light is passed through. In the Wagner Turbidimeter or similar types of apparatus a photometer is placed behind the chamber, which measures the light transmitted through the turbid suspension. In the Tyndallometer the photometer is placed at right angles to the path of the beam and the amount of light dispersed by the suspension is measured.

The formulae used to convert the accurate light measurements into surface values are dependent upon an empirical constant which must be evaluated by some other method. For material below 5 microns in diameter the formulae do not apply since the particle size is too close to the wave length of light. Fundamental theory is of little use since the optical relationships are not well enough known to permit of mathematical analysis.

The method is rapid since only a few seconds are required to make the photometer measurement. Results can be calculated directly by formula without size being determined. Reproducibility on a given sample is excellent. Therefore the method is used extensively for routine determination of cement fineness and is designated as an A.S.T.M. Standard(31). Standard Turbidimeter cement samples are obtainable from the U.S. Bureau of Standards.

Details of apparatus, technique and calculations, are given in the A.S.T.M. Standard as well as by Taggart[5-126], Dallavalle[11-272], and Pieters and Hovers[21]. A thorough treatment of the theory is given by Rose and Lloyd[32] [33].

However due to the uncertain relationship between translucence of material and light interference turbidimetry measurements of surface can be considered as relative only. This is shown by the tabulated comparisons between turbidimeter results and surface values by other methods given by Roller and Roundy[25], Pieters and Hovers[21], and Lea and Nurse[34].

Coercimetry Method

The coercimeter is an instrument used to measure the coercive force, that is, the force of retention of magnetism, of a sample of material. It has been shown by Gottschalk[35] that a linear relationship exists between the coercive force and the specific surface of magnetite. Since the coercive force can be quickly measured, the method can be used to determine the specific surface of magnetite and presumably any other magnetic material.

The apparatus required is: (1) a strong magnetising device, and (2) a coercimeter apparatus. Apparatus is described in detail by Gottschalk[36-21], and details of operation by Wartman[36-33]. The underlying theory is discussed thoroughly by R.S. Dean[36-3].

Coercimetry for surface determination is applicable only to magnetic materials, in particular magnetite, since its use on other magnetic materials is undeveloped. It requires intricate and accurate electro-magnetic apparatus.

The accuracy of the determination appears to be very sensitive to the method and extent of packing of the powder,particularly with fine sizes[36-38].

Permeability Method

The permeability method is based on the assumption of a definite physical relationship between the specific surface of the powder and the permeability of the powder to fluid flow. That this is approximately true has been shown by many research workers, but the exact mathematical relationship is as yet not fully determined. The relationship is influenced by several other factors: porosity, uniformity of packing, shape of particles, and nature of fluid flow. Empirical solution of the effect of these factors has reduced the errors possible, so that the method is at least as accurate as any other surface determination method. However, in spite of the inaccuracy of absolute surface values, the method gives very good reproducibility. This has been shown by many investigators to be about 2 per cent.

The method has several other advantages over other direct methods:

(1) Adaptability to any homogeneous or heterogenous powder, of any degree of fineness.

(2) Simplicity of apparatus.

(3) Simplicity of operation.

(4) Extreme rapidity.

(5) Simplicity of calculations.

Therefore the method is adaptable to routine testing and is finding an increasing number of uses in the cement, coal, pigment, fibre and food industries.

The apparatus can be adapted to use any fluid, liquid or gaseous, depending on the nature of the powder and the conditions of the test. Air appears to be the most convenient for normal powders.

A limitation of the fluid permeability method is the quantity of sample required. At least one gram would seem to be the minimum for accuracy in the present types of apparatus. Also the method measures external surface only, and takes no account of cracks and pores.

Since this method forms the basis of the experimental work in this thesis, detailed description of apparatus and discussion of theory will be given in a separate section dealing with the development of the Air Permeability Method. From the detailed discussion in this separate section, the validity of the above claims of superiority can be assessed.

Other Methods of Direct Determination of Surface

Other methods of surface determination have been developed which are only applicable to specific powders.

An example is the determination of specific surface of carbon black by making a smear on a glass slide and comparing the intensity of color with known standards(37).

Summary

Direct methods of surface determination are at least as accurate as indirect methods, with the advantage of superior speed and reproducibility. An appropriate direct method can be chosen to fit any powder of any fineness, to measure either external or total surface as required. The surface can be measured in any condition, wet or dry, by some direct method. Direct methods are particularly adaptable to routine testing where relative values of good reproducibility are required quickly.

No direct method can be said to give an absolute value for specific surface due to the physical assumptions necessary, but experimental evidence shows that direct methods are sufficiently accurate for any practical use in control of industrial products. With the current extension of fundamental research, some of the methods may be modified to give very accurate results.

Dr. Heywood(38) states that the three most promising methods for determining the fineness of a powdered material are the adsorption method, the permeability method and the turbidimeter method.

Without some dependable method of determining the state of subdivision of powdered material, many modern industries dependent upon powdered material are seriously hampered. Direct measurement of specific surface appears to be the most practical solution to the problem.

SUMMARY

From the consideration of the literature dealing with the various methods of specific surface determination, and particularly the air permeability method, the following conclusions may be drawn:

(1) Direct methods are preferable to indirect methods since the effects of particle size and particle shape are minimized.

(2) Of the direct methods, the air permeability method has the greatest range of applicability, coupled with ease of operation, rapidity, and simplicity of apparatus.

(3) The air permeability method is particularly adaptable to routine testing due to its good reproducibility.

(4) Calculation of results by formula is not as accurate as the reproducibility of the apparatus. Therefore the method is best used

empirically with a suitable standard as reference.

(5) Spherical powders which can be accurately sized by microscope provide the only accurate standard.

Therefore the air permeability apparatus calibrated with a spherical powder provides the ideal apparatus for determining the specific surface of any powder, the accuracy being limited only by the reproducibility of the apparatus.

In evaluating milling efficiency further information is frequently required. Not only must the total surface be known but the distribution of that surface between the various size grades and component minerals is important. This involves preliminary fractionation of the material which can be done most accurately by elutriation. Rising current elutriation, either by water or air, is the simplest type of elutriation.

For a heterodisperse powder, elutriation ~~will~~ yields the required range of fractions. A specific surface determination on each fraction ~~will~~ gives the distribution of surface for the powder.

For a heterogeneous powder the same information is obtained. However if the powder is essentially composed of a few minerals which can be quantitatively determined by a chemical analysis, the distribution of surface among the minerals, as well as the relative size of the minerals in each fraction, can be obtained by elutriation and surface determination. A preliminary calibration must be made by elutriation and specific surface determination on each of the pure minerals. Then this data can be applied to the results of a test on any mill product containing the minerals.

Three important applications of the calibrated air permeability apparatus are therefore possible:

(1) Determination of the specific surface of any mill product directly and rapidly.

(2) Determination of the distribution of specific surface in any homogeneous mill product by correlation with standardized elutriation.

(3) Determination of the distribution of specific surface amongst the component minerals by correlation with standardized elutriation and chemical analysis.

In order to confirm the practicability of these determinations, the experimental work, to be described, was undertaken.

THE AIR PERMEABILITY METHOD

Of the direct methods of specific surface determination the method most readily adapted to the normal range of finely ground materials is the Air Permeability Method. The reasons for this are given in the preceding section and will now be explained in detail.

The basis of the method is the physical relation between fluid flow through a porous bed and the specific surface of the material in the bed.

In general the method is as follows:

(1) The powder to be measured is compacted into a bed of uniform known porosity in a small cell.

(2) Air is passed through the cell under a known pressure head.

(3) The rate of flow of the air is measured.

(4) The specific surface is evaluated either by calculation from formulae, or by calibration curves.

A description of the apparatus used and its development from the original liquid permeability apparatus will be followed by discussion of the theoretical considerations.

APPARATUS

The air permeability method was developed from the original liquid permeability method. The use of liquid, although giving good results, was restricted by physical factors. Therefore gaseous fluids were substituted.

The main consideration in favor of the liquid method is the simplicity of apparatus. The principle problem in the design of air permeability apparatus is to produce equally simple, yet accurate, flow measurement apparatus.

Liquid Permeability Apparatus

The original apparatus for determining specific surface by fluid permeability was the liquid permeability apparatus developed by Carman[39][40]. Since it is still used as a reference method and is the basis of all subsequent air permeability methods, a description of Carman's apparatus and method will form a foundation for discussion of the newer methods.

The bed of powder is supported in a glass tube on a piece of monel metal filter cloth. As the filter cloth will not support fine material, a thin supporting layer of coarse material is built up on the cloth by depositing a layer of 150 micron sand, topped by a layer of Kieselguhr. The total thickness of this supporting bed is about 3 millimeters, and its resistance to flow must be determined, and added to the result of permeability measurements.

The sample is pulped with the liquid to be used and is deposited by suction on the prepared layer in the cell. The depth of the bed is then measured.

The cell is then placed on the apparatus and the measurement of permeability is made. The liquid is fed into the top of the cell by a delivery tube from a measuring cylinder, so that the free surface of the liquid in the cell is maintained at a constant level. The liquid is drawn through the bed into a suction flask, the required suction being maintained by a vacuum pump and pressure regulator, and measured by a manometer. Additional driving force is obtained from the head of liquid between the level in the cell and the discharge into the flask, so this must be measured. The total driving pressure is calculated. The suction flask is graduated so that the rate of flow of fluid through the

bed is measured for a run of one hour.

Calculation of specific surface is made using the formula:

$$S_o = 14\sqrt{\frac{1}{K_1} \cdot \frac{E^3}{(1 - E)^2}} \quad ,$$

where K_1 is the permeability factor and equals

$$\frac{Q\ U\ L}{(P_1 - P_2)\ A} \ .$$

Q = rate of flow (ml. per second).

U = viscosity of the fluid (poises).

L = depth of the powder bed (cm.).

A = area of powder bed (cm.^{2}).

$P_1 - P_2$ = total driving pressure less the correction for the supporting bed (gm. per cm.^{2} of packing).

E = fractional porosity $= 1 - \dfrac{W}{d_1\ AL}$

W = wt. of powder (gm.).

d_1 = density of powder (gms. per cm.^{3}).

Derivation of this formula will be covered in the section on theoretical considerations.

Carman's method is very simple and requires only one special piece of apparatus - the permeability cell. A vacuum system capable of maintaining a uniform, constant vacuum for an hour is required.

The method has several disadvantages however:

(1) The preparation of the bed results in non-uniformity of the bed, since lamination is produced with unsized material.

(2) The liquid must be carefully selected to avoid flocculation and reaction between the material and the liquid.

(3) With fine powders adsorption of an immovable layer of liquid on the solid surface effectively alters the porosity.

(4) With fine powders flow is so slow that excessive time must be used or inaccuracy of volume measurements results.

However the method is rapid for coarse powders. As used by Carman it gave very good results. The air permeability method, developed by Lea and Nurse and subsequent investigators, is much superior.

Air Permeability Apparatus

The air permeability apparatus originated from experiments on the use of air as the fluid in Carman's apparatus and is based on the same theory.

Air Permeability apparatus can be classified into three types, depending on the method of measurement of flow rate:

1. Volume-flow apparatus.
2. Instantaneous-rate-of-flow apparatus, of the differential manometer type.
3. U-tube manometer types based on either volume-flow or instantaneous-rate measurement.

Volume-Flow Apparatus: The original apparatus used by Lea and Nurse(34) is fundamentally the same as Carman's with an improved design of the permeability cell and the addition of apparatus for measuring air flow.

The cell is made of two short, flanged metal cylinders which hold a perforated brass plate when bolted together as in Figure 1. The sample is compressed dry into the upper half by use of a metal plunger with an adjustable shoulder to gauge the depth of bed.

The air flow apparatus consists of a large jar from which water is drained at a constant rate, drawing air through the cell which

is connected to the top of the jar. Manometers indicate the differential pressure across the bed. Normal flow rate is 1 to 4 litres of air per hour through a bed of 1 inch diameter and approximately 2 inches in depth. Calculations are the same as for the Liquid permeability method.

The apparatus with some modifications was used with good results by J. Oberholtzer to determine the specific surface of powdered coals.(41)

However the apparatus is cumbersome and time for a test is excessive, particularly with fine samples. Therefore, recent investigators have modified the Lea and Nurse method to make the present apparatus more compact and to shorten the testing time.

Differential Manometer Apparatus: The typical apparatus of the differential manometer type is that of Gooden and Smith(42)(5-135). A commercial model is manufactured and sold for cement testing in America(43).

In this type, air is either drawn or forced through a cell containing the compacted sample by a constant pressure or constant vacuum system. A pressure regulator of some type controls the pressure on one side of the bed. The air flowing through this system passes through a constriction across which is connected a differential manometer. The manometer indicates the rate of flow through the constriction. By this method only the time for the rate of flow to become constant is required. This is normally only a few minutes. Gooden uses a special method to compress the powder into the cell which gives uniform porosity with little time(44). Calculations are simplified and set up in a special alignment chart.

The apparatus, with the exception of the constant pressure equipment, is simple and can be set up in a small, portable unit. Good reproducibility is obtained, and with composite samples good checks are obtained[42].

The apparatus has the disadvantage of requiring auxiliary pressure or vacuum equipment. Satisfactory equipment is not always obtainable, which places a limitation upon the method.

U-tube Manometer Apparatus: Air Permeability apparatus of manometer type operating on the mechanical principle of the Ostwald viscometer, was developed simultaneously by J.P. Rigden in England[45], and by R.L. Blaine in the U.S.A.[46]. The two methods are very similar but the differences, although slight, are sufficient to affect the application of the methods.

The Rigden Apparatus, shown in Figure 1, has been used by Rigden[45] on material ranging from silica powders with specific surface of 1000, to fine cements and powders with specific surface of 10,000.

The apparatus consists of two large-bore vertical tubes, joined at the bottom to form a U-tube. The tubes are connected at the top through the permeability cell containing the powder. This connection can be closed by a stop cock. The U-tube is filled to the half-way mark with a liquid of low viscosity and low vapour pressure. The two side arms, one at the top of each large tube, are fitted with stop cocks. In the original apparatus two marks are placed on the left tube, one, A, is 3 centimeters above the equilibrium level, and the other, B, is 10 centimeters above the equilibrium level.

In operation, the sample is placed in the cell which is the Lea and Nurse type, and is compacted in increments to a uniform known

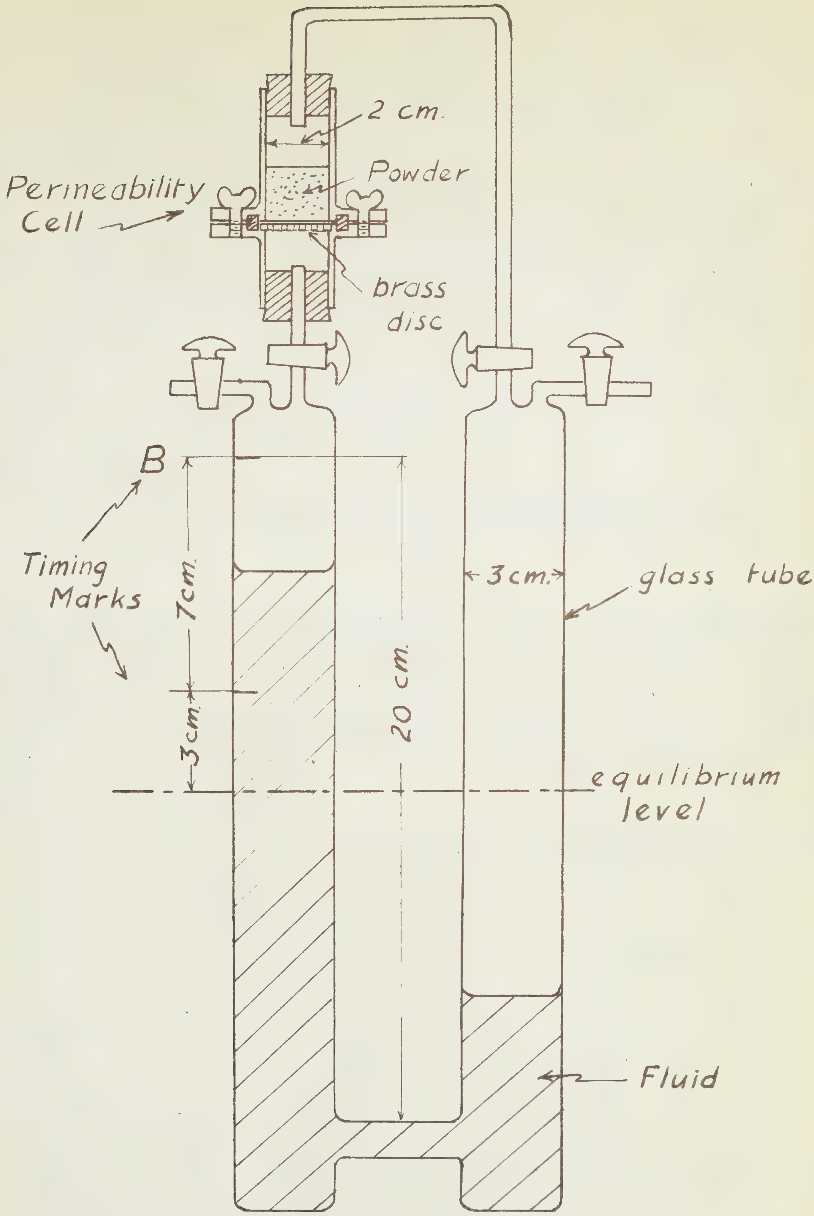

FIG. 1 - RIGDEN AIR PERMEABILITY APPARATUS

porosity, each increment being compacted by tapping with the plunger, and weighed to ensure uniformity. The cell is then connected into the cross tube. The cross connection is closed by the stop cock. With the right side-arm connection open, suction is applied to the left side connection to draw the liquid up to a level in the left tube about 1 centimeter above the mark B. Side connections are then closed.

The cross connection is then opened and the liquid, falling to equilibrium in the U-tube, draws air through the sample cell. The time required for the level in the left tube to fall from B to A is taken by stop watch.

The specific surface is given by the equation,

$$S = K\sqrt{t},$$

where K is calculated from the dimensions of the tube, viscosity and density of the liquid, viscosity of air, and porosity of the sample bed. The derivation of the formula and calculations will be discussed in the section on theory.

It is evident that the apparatus is completely self contained and entirely independent of auxiliary equipment. This is its main advantage over other types. Also only a single measurement of time is required to obtain the specific surface of a powder, providing that the porosity and bed depth can be standardized. The measurement is independent of barometric pressure. Corrections for temperature are compensating, drop in viscosity of ~~air and~~ liquid with higher temperature being compensated for by expansion of the liquid in the U-tube and corresponding lowering of the driving head.

The apparatus can be adapted to any range of powders by modifications which will give a measureable time interval which ensures

viscous flow of air through the sample. Slight variations can be allowed for by altering the depth of the bed in the cell. Greater changes in range may require a change in the diameter of the sample cell or a change in the diameter of the U-tube limbs.

Assuming the validity of the theory and the formula, this apparatus gives the most convenient and rapid method of obtaining specific surface of a powder and is particularly valuable for routine testing.

The apparatus used by Arnell[8] is a modification of the Rigden type. With very low permeability, Rigden's simple equation is not correct since the rate of change of pressure through the bed is not a linear function. An equation can be derived which will allow for this, but calculation of results becomes very cumbersome. Arnell has found that measurement of the differential pressure across the bed provides data from which the rate of flow can be calculated without any further measurements.

By use of a reference manometer and a measuring manometer connected to the Rigden apparatus, the pressure across the bed can be measured at any time during the fall of the liquid. From readings at two positions above the equilibrium level, the rate of flow and the specific surface can be calculated. Arnell has used this apparatus for measuring specific surfaces as high as 4.6×10^5 corresponding to theoretical particle diameters of 0.2 microns.

The Blaine Apparatus[46][47] is a simple U-tube of large bore. To the upper end of one limb are attached a side arm and the permeability cell. The cell is made from a section of glass tubing, and the powder bed is supported on a perforated disc ground to fit on a ledge inside

the cell. The U-tube is half filled with a liquid of low viscosity and low vapour pressure. Timing marks are placed on the tube as for the Rigden Apparatus.

To make a surface determination the powder is compacted in one operation in the cell by means of a bakelite plunger with a shoulder which gives a fixed bed depth. The weight of powder to give the desired porosity at this depth is predetermined for the material being tested. After compaction of the powder the cell is placed on the U-tube and time for flow, t, measured as for the Rigden apparatus. Calculations are by the formula:

$$S = K\sqrt{t}$$

where K is a constant for the apparatus.

The method is rapid since the apparatus is designed so that t is from 2 to 5 seconds. Calculations are simple. No special technique or training is required. The apparatus is entirely self contained, easily constructed, and portable.

The Blaine method was checked by 18 cement testing laboratories under the direction of the A.S.T.M. In the report(48)(49) very good agreement between the different laboratories is shown, although the calculation formula used is shown to require some modification. Therefore the method is proposed as an A.S.T.M. Standard for Testing Cement(47).

The disadvantage of the Blaine apparatus is its restricted range due to small air volume. This limits the range of the apparatus to powders of specific surface above 1000. This range, however, includes all cements, pigments, and most soils(54).

Summary

The development of the method from early measurements of permeability to the present testing method is typical of the evolution of

a new scientific technique. At present although patented apparatus of reasonable accuracy can be purchased, fundamental research into the various physical factors involved is being carried out by several investigators and new and more accurate apparatus is being developed.

However, the development of apparatus has proceeded at a faster rate than the development of the theory, so that the apparatus is now far more accurate than the equations which are used to convert the experimental data to a specific surface value.

In the following section, the basic theory will be discussed, and the equations at present in use will be derived.

THEORY OF AIR PERMEABILITY

As stated above, several factors enter into the calculation of specific surface from the air permeability data. Of these the most important are: porosity of bed, shape of particles, orientation of particles, and type of flow.

The equation for fluid flow through a porous bed has been the subject of much research and much controversy for many years. Recently this subject has received increasing attention due to the development of the air permeability method for surface determination.

Basic Flow Equation for Air Permeability

Basic formulae for fluid flow through porous beds are similar but differ as to derivation and assumptions. Dallavalle[11-X] gives several formulae and discusses their applications.

The formula most used in the fluid permeability method for surface determination is known as the Kozeny formula. It is based upon Darcy's Law which gives the relationship between rate of flow and pressure

drop through a porous bed as:

$$Q = P_o\, A\, \frac{p_1 - p_2}{L} \qquad \text{Eqn. (1)}$$

where $p_1 - p_2$ = pressure drop

L = length of packing

A = cross section area of packing

p_o = permeability constant depending on the fluid, temperature and packing.

If the viscosity of the fluid is U, Darcy's equation becomes:

$$Q = \frac{P}{U} \cdot A \cdot \frac{p_1 - p_2}{L}$$

where P is the permeability of the packing, in darcies. This equation assumes change in pressure along the packing to be constant. The more general form where change in pressure is variable is:

$$Q = \frac{P}{U} \cdot A \cdot \frac{dp}{dx} \qquad \text{Eqn. (2)}$$

From experimental evidence Poiseuille derived the Poiseuille equation for flow in pipes:

$$Q = \frac{p_1 - p_2}{L} \cdot \frac{r^2 A}{8\,U} \qquad \text{Eqn. (3)}$$

This follows Darcy's Law if $P = \frac{r^2}{8}$ for pipes.

Poiseuille's equation can be generalized for tubes or capillaries of any cross section if the radius of the cylindrical pipe, 'r' is replaced by a general linear dimension characteristic of the cross section of the pipe. If the linear dimension chosen is the mean hydraulic radius 'm' and a flow constant 'k_o' for the shape of cross section is known, the equation for flow through a straight

channel of any shape is:

$$Q = \frac{m^2}{k_o U} \cdot A \cdot \frac{p1 - p2}{L} . \qquad \text{Eqn. (4)}$$

For circular pipes of uniform cross section

$$m = \frac{\text{volume of tube}}{\text{surface of tube}} = \frac{\text{area of tube section}}{\text{circumference}} = \frac{r^2}{2\ r} = \frac{r}{2} .$$

Substituting in the Poiseuille equation for circular pipes,

$$Q = \frac{r^2 A}{8U} \cdot \frac{p_1 - p_2}{L} = \frac{(2m)^2 A}{8\ U} \cdot \frac{p_1 - p_2}{L} = \frac{m^2 A (p_1 - p_2)}{2\ U\ L} .$$

Therefore for circular pipes $k_o = 2$. For triangular cross sections k_o equals 1.67, and for rectangular cross sections, of breadth ten times the height, k_o equals 2.65.

Equation 4 has been found to hold for ~~any~~ straight channels of any cross section. The constant 'k_o' must be calculated or determined experimentally for each case.

For the basic mathematical treatment of flow through porous beds, Darcy's Law is also assumed. The Poiseuille equation for straight tubes is adapted to the tortuous channels in a porous bed. The mean hydraulic radius for the channels through the bed is derived as follows:

By definition $m = \frac{\text{volume of conduits}}{\text{surface of the conduit}}$. Therefore

for porous beds $m = \frac{\text{volume of void space}}{\text{surface of the particles}}$

$$= \frac{E \times \text{volume of bed}}{S\ d_1\ (1 - E) \times \text{volume of bed}}$$

$$= \frac{E}{S\ d_1\ (1 - E)} ,$$

where S = specific surface

E = fractional voids = porosity

d_1 = density of material.

The total area of the channels becomes AE where

A = area of the bed.

Due to the tortuosity of the pores the length of the channels must be increased by a factor so that the mean length of the channels becomes $\frac{L}{y}$, where y is the length factor.

Substituting these values in equation (4)

$$Q = \left(\frac{E}{Sd_1(1-E)}\right)^2 \cdot A\,E \cdot \frac{p_1 - p_2}{k_o\,U\,\frac{L}{y}}$$

$$= \frac{A}{s^2 d_1{}^2 k_o} \cdot \frac{y(p_1 - p_2)}{U\,L} \cdot \frac{E^3}{(1 - E)^2} \quad \text{Eqn. (5)}$$

This is the basic equation for flow of fluids through a porous bed. Since it involves the specific surface it is also the basic equation for the fluid permeability method of surface determination.

The Kozeny equation as normally used is

$$Q = \frac{A}{s^2\,d_1{}^2\,k} \cdot \frac{p_1 - p_2}{U\,L} \cdot \frac{E^3}{(1 - E)^2},$$

where $k = \frac{k_o}{y}$.

Kozeny found k = 5 for porous beds, and this value was checked by Carman(40).

Due to the assumptions involved in the derivation of the basic equation, it is not surprising that investigators have found that it is not strictly accurate. Evidence has been obtained to prove that the channel shape factor 'k' of Carman and Kozeny is not constant for different types of packing. The validity of the porosity function is questionable over any large range of porosity. In the original derivation it is assumed that $\frac{dp}{dx}$ is constant for the length of the bed. Actually this is only approximately true for limited ranges of pressure difference(50). Also it has recently been proven that in addition to mass flow of gas, molecular diffusion or slip takes place, so that the

finer beds require a correction to the basic equation. Therefore the calculated results, using the Carman-Kozeny equation are appreciably in error.

The factors which tend to modify the basic equation will be discussed in detail in the following sections.

Effect of Channel Shape

The channel shape factor 'k' is introduced to correct for the effect of channel cross-section on the flow, and to account for the increased mean length of the channels due to the tortuous path which the fluid must follow in passing through a bed of particles. It is therefore considered as equal to $\frac{k_o}{y}$ where k_o is a factor affected by channel cross-section and y is a factor affected by channel tortuosity[51]. Both factors are influenced by the shape of the particles, the orientation of the particles, and the porosity.

As stated above k_o varies for straight channels of regular geometric cross-section. For straight channels through a porous bed made up of cylindrical rods, the value of k_o was found experimentally by Emmersleben[51]. Using equation 5 he found k_o equal to 3 with porosities below 0.84.

The effective shape of a channel formed between a number of particles is difficult to determine and varies with the nature of the packing. This characteristic of packing is discussed by Dallavalle[11-VI]. In experiments on particles of circular cross-section, Sullivan and Hertel found k = 3.07 for beds of glass fibres oriented parallel to the flow, k = 4.50 for beds of spheres when E = 0.39, and k = 6.04 for beds of fibres at right angles to the flow. From theoretical

considerations $y = 1$, 0.66, and 0.5 for these cases so that $k_o = 3$ for all cases. Also for random oriented irregular particles Sullivan and Hertel found $k = 4.50$, and using a theoretical value of $y = 0.66$, $k_o = 3$. Therefore for spherical, cylindrical, and random oriented irregular particles, k_o appears to have a value of 3.

However Carman found that $k = 5$ over a range of particle shapes including glass spheres, and ground silica; Muskat and Bottsett[51] found $k = 4.65$ for glass spheres; and Fowler and Hertel found $k = 5.5$ for beds of fibres. Also Muskat and Botsett found that in a piece of sandstone flow perpendicular to the bedding was only 70 per cent. of flow parallel to the bedding. Sullivan and Hertel[51] postulate that these variations are due to variations in y, since as shown above, y varies considerably with the orientation of the particles. Since Carman's beds were prepared by sedimentation there would be a tendency for preferred orientation of particles perpendicular to the axis of the bed. This would give a lower value for 'y' than in the dry packed beds of Bottset, and Sullivan and Hertel. Assuming the constant value of 3 for k_o, the value of $k = \frac{k_o}{y}$ would be greater for Carman's beds. This would also explain the anomalous flow in the sandstone.

Carman[52] in discussing the slip correction for fine powders shows that the normal value for k_o appears to be 2.5, when molecular diffusion through packings is considered. It is not clear whether this value also applies to mass flow or is limited to slip flow.

It is generally agreed that until more fundamental research has been done into the effect of particle shape, packing and orientation, the experimental work of Carman warrants the use of Carman's value of 5 for k for flow through the normal irregular shaped powders, where a

certain amount of preferred orientation perpendicular to the axis of the bed is produced in packing the bed. In special cases of spheres at low porosities, packings of fibres, or other particles of abnormal shape or known orientation, the specific values of k for those packings should be determined and used.

Effect of Porosity

The subject of porosity of powders and its effect on flow of fluid through porous packings is discussed at length by Dallavalle (11-VI)(11-X). The packing of spheres is treated in detail and the various corrections proposed for non-spherical powders are given. It is evident that there is no definite arrangement of particles, and formulae for porosity are only empirical. Very little is known of the nature of the void spaces. With very fine powders there is evidence of repulsion of particles within the packing and of cushioning of particles by adsorbed layers of gas[43]. These effects introduce the consideration of "effective" porosity, which may have no relation to actual porosity when fluid flow is considered.

The porosity of a packed bed of powder is allied with the shape, packing and orientation of the particles. Its effect on fluid flow is therefore a very complex problem and the nature of the function relating porosity to flow rate has been the subject of much investigation.

The effect of shape of particles on porosity is indicated by the experiments on spheres and cylindrical fibres conducted by Sullivan and Hertel and Fowler and Hertel[51]. They found that the normal porosity for beds of glass and textile fibres, without deforming the fibres, was about 0.80. On the other hand for beds of spheres the normal porosity is about 0.40 with the theoretical closest packing for

even sized spheres giving a porosity of 0.26(11-105). The theoretical most open packing for spheres is 0.476. Gratton and Fraser prepared a table(11-121) showing the influence of particle shape on porosity of a number of materials. Dallavalle(11-V) gives a thorough discussion of the relation between particle shape and packing and void space.

The effect of porosity on pore diameter is also discussed by Dallavalle(11-V). Traxler and Baum(11-119) obtained an emperical equation relating pore diameter and porosity:

$$\ln D_c = b + 0.019 E ,$$

where D_c = mean diameter of pore

E = porosity

b = a constant which must be determined for each material.

The effects of increase in porosity on fluid flow are due to: (1) increase in the effective free area or total channel area in any plane perpendicular to the axis of the packing, and (2) decrease in the length of the path followed by the fluid in passing through the packing.

The effective free area, 'a', for spheres, ranges from 9.3 to 21.5 per cent.(11-109) depending on the mode of packing. Traxler and Baum give the relation between a and E as:

$$\ln a = KE + c .$$

Substituting values for a and E, measured by Hodgins, Flood and Dacey(50) on a sintered glass plug, K = 1.41, and c = -1.40, so that

$$\ln a = 1.41 E - 1.40 .$$

This equation, solved for closest and most open packings for spheres, gives values of 0.093 and 0.180, in good agreement with the values of

9.3 and 21.5 per cent. obtained experimentally by Dallavalle.

Arnell[8] shows that the effective free area factor 'a' should be used as the correction for the total bed area in deriving the basic flow equation, rather than the porosity 'E'. This gives the effective total channel area of bed as 'aA'. The basic equation then becomes:

$$Q = \frac{A}{s^2 \ d^2 \ k_o} \cdot \frac{p_1 - p_2}{U} \cdot \frac{y}{L} \cdot \frac{E^2 \ a}{(1 - E)^2} .$$

This amended equation shows therefore that the rate of flow should vary as $\left(\frac{E}{(1 - E)}\right)^2$ and also as 'ya' where 'y' and 'a' are both dependent upon the porosity and mode of packing. It appears certain that the rate of flow will not vary directly as Kozeny's $\frac{E^3}{(1 - E)^2}$, except in cases where for narrow ranges of 'E', both 'a' and 'y' vary directly as 'E'.

That the porosity function is even more indefinite than indicated above is shown by the variety of functions which have been accepted and can be proven from theoretical assumptions. In addition to the two functions given above, Buckingham proved that 'Q' varied as $E^{7.0}$, Slichter proved 'Q' should vary as $E^{3.3}$, and Mavis and Wilsey proved 'Q' varied as $E^{6.0}$ (11-207).

It seems therefore that the relationship between rate of flow and porosity does not submit to exact mathematical treatment. Although the Kozeny and Arnell equations give close approximations to experimental results they are not sufficiently accurate for extrapolation over ranges of porosity and particle shape. An emperical relationship is therefore more useful.

Emperical equations based upon actual experimental evidence have been evolved by Chilton and Colburn[11-208], and by Furnas.[53] Furnas' equation is considered the best available for treatment of gas flow through broken materials at high temperatures. These equations are applicable only when the constants required for their solution have been predetermined for the particular application.

Effect of Slip Flow

The above derived equations give approximate values at normal porosities and for powders of specific surfaces up to 2000. However it was noticed by several investigators[55] that there was a serious disagreement between values of specific surface by liquid permeability and values obtained on the same powders by air permeability. This was particularly evident on cements with specific surface above 2000. Originally the disagreement was blamed on an immobile layer forming at the solid - liquid interface and reducing the effective porosity. Recently fundamental research has proven that the higher rate of air flow in the fine capillaries of fine powders is due to molecular slip.

The physical nature of slip, and the application of slip flow equations is treated by Rigden[55][56], Hodgins, Flood and Dacey[50], Nissan[57], Arnell[8], and Carman[52]. Two different approaches to the subject are given, one thermodynamic, and the other physical. The results are equations which give air permeability specific surfaces in good agreement with the liquid permeability values.

Arnell[8] gives a mathematical treatment based on Knudsen's equation for molecular flow of a gas when the mean free path of the molecules is large compared to the capillary diameter. His final

equation is:

$$Q = \frac{h\, d_1\, g\, A}{k\, S_v^2\, UL} \cdot \frac{E^3}{(1 - E)^2} + \delta\, \frac{8}{3}\sqrt{\frac{2}{\pi}}\ \sqrt{\frac{R_o T}{M}}\ \frac{\Delta p}{PL} \cdot \frac{a\, A\, E}{S_v\, (1 - E)}$$

where the first term is the basic Kozeny equation for the Ridgen apparatus, and the second term is derived from Knudsen's equation.

In this equation M = molecular weight of the gas

T = absolute temperature

R = gas constant

Δp = pressure difference across the bed

P = mean pressure across the bed

a = effective void area

S_v = surface of 1 cm^3 of packing

δ = a factor = 0.9 approximately.

This equation was tested by Arnell on specially designed apparatus over wide ranges of pressure, porosity and specific surface and found to hold. It was used with a special Rigden type apparatus for measurement of specific surface of very fine pigments and powders of specific surface up to 4.6×10^6.

Carman(52) gives the derivation of a flow formula based on Millikan's equation for slip velocity at a capillary wall. This equation takes the form of a correction to the basic Kozeny equation where the actual flow velocity, u_o, equals the velocity of flow, u, from the Kozeny equation plus the velocity of slip from Millican's equation. That is,

$$u_o = u\ (C)$$

where C is a correction factor and equals:

$$1 + \frac{S\, d_1\, (1 - E)\, z\, \lambda}{E}$$

λ = mean free path of molecules

d_1 = density of the solid material

S = specific surface

$z = k_o \left(\frac{2}{f} - 1\right) = 2.73$ to 4.07 (average 3.3)

where k_o = 2.5 to 3

and f = 0.84 to 0.89

= fraction of molecules striking and reflected from the capillary walls.

Lea and Nurse are reported to have used this equation for fine powders(52) but their results are not yet published.

Rigden(56) derives the equation

$$v = \frac{A\ p\ g\ E}{k\ U\ L} \cdot \left[\frac{1}{S^2} \left\{ \frac{E}{(1 - E)\ d_1} \right\}^2 + \frac{1}{S} \cdot \frac{2E\ f\ \lambda}{(1 - E) d_1} \right]$$

where k = Kozeny's constant = 5 (approx.)

f = 0.874 for air, and where other symbols are as for Carman' equation. This gives a correction slightly lower than the Arnell or Carman equations.

It is evident that even if these modified equations give approximately correct results, they are very cumbersome to use for surface determination since the correction term involves the specific surface. Therefore empirical relationships would seem to be preferable.

The correction equation used by Carman, and Lea and Nurse, is used in this investigation in comparing the empirical calibration curves with theoretical values. The equation must be put into a workable form suiting the apparatus used and the data obtained.

ACCURACY OF THE AIR PERMEABILITY METHOD

The theoretical treatment of the air permeability method yields equations of doubtful accuracy unless the use of the equations is confined to ranges of materials and porosities for which the constants have been evaluated with reasonable accuracy. Used empirically for ranges where the equations are proven the method is reproducible within 5 per cent.

Reproducibility

This reproducibility has been demonstrated by many investigators. Rigden[56] gives an analysis of systematic and random errors for the U-tube apparatus and derives a possible maximum error of 3.9 per cent. The standard deviation of his actual experimental values varied from 0.5 to 2.7 per cent. for different powders. The A.S.T.M. report on tests made on the Blaine apparatus[48] states that the average variation was only 1.1 per cent. for eight operators making five determinations on each of four cements ranging in specific surface from 2490 to 5130. Lea and Nurse[34] obtained reproducibility within 2 per cent. on cements and silica sands. Gooden and Smith[42] were able to check on the specific surface of mixtures within 5 per cent. Carman obtained reproducible results within 5 per cent. for powders down to 2.0 microns in diameter.

Absolute accuracy

The absolute values of specific surface as determined using the Kozeny equation are not to be considered accurate within 5 per cent. even on spherical samples[52]. Arnell using his modified equation claims accuracy within 10 per cent. for very fine powders in checking

against electron microscope results.

Comparison with Other Methods

Claims of agreement with other methods are made. Lea and Nurse found very poor agreement with other methods[34]. Of these the turbidimeter method is known to give low values, the gas adsorption method gives high values, and the microscopic method is dependent for accuracy upon an assumed shape factor. Therefore agreement or lack of agreement with results of other methods is not in itself indicative of accuracy.

However the relation between air permeability results and the results of any other method appears to be constant. Lea and Nurse found a constant factor relating air permeability and turbidimeter results[34]. A similar relationship was found by Blaine[46].

Summary

Therefore as an analytical instrument for accurate determination of absolute values, the method may or may not be more accurate than other methods. However, as a tool for process control where reproducible relative values are required, the air permeability method is superior.

EXPERIMENTAL INVESTIGATIONS

Preliminary studies of the air permeability method indicated that it could be satisfactorily applied to specific surface determinations on any ground product if calibrated accurately. Accordingly investigations were carried out for the purpose of arriving at a suitable design for an air permeability apparatus and a suitable method for its calibration. Throughout the work the main object was to develop apparatus which could be used to obtain reasonably accurate measurements under industrial testing conditions.

CALIBRATION OF THE AIR PERMEABILITY APPARATUS

The calibrated air permeability apparatus was obtained by the following procedure:

1. Preparation of surface standards
2. Design of the Air Permeability Apparatus
3. Preparation of Calibration Curves.

Tests on the completed, calibrated air permeability apparatus indicated that its accuracy was well within the limits desired.

PREPARATION OF SURFACE STANDARDS

A suitable surface standard for calibration of an air permeability apparatus is a powder composed of glass spheres whose specific surface can be determined accurately by microscopic methods.

The preparation of a series of standard spherical powders covering the range of specific surface values desired involved three stages:

1. Preparation of microscopic glass spheres.

2. Sizing of the spheres by elutriation.

3. Microscopic evaluation of the specific surface of the sized fractions.

Preparation of Microscopic Glass Spheres

Microscopic glass spheres have been prepared by Bloomquist and Clark of Battelle Memorial Institute[19], Pieters and Hovers[7], and others. Sized spheres from 3.3 to 25 microns in diameter can be purchased from Battelle Institute. However for calibration of the air permeability apparatus, larger spheres ranging up to 74 microns were desired. Therefore it was decided to prepare the spheres in the laboratories of the Department of Mining.

The procedure is briefly as follows: Glass powder is fed into the air stream of a blast type burner. The powder fuses in the burner flame, is drawn spherical by surface tension, and leaves the flame as glass spheres. Collection apparatus recovers the product.

Apparatus: Preliminary work on sphere preparation was carried out during the 1945-46 term by Lauer and Chalmers, but redesign of the apparatus was necessary to increase sphericity, and improve collection and recovery. After considerable experimentation a satisfactory apparatus was designed which could be controlled to yield a uniform product of good quality. The component assemblies whose design and operation will now be described are:

1. The control panel.
2. The powder feeder.
3. The burner.
4. The collection system.

The arrangement of these components to form the complete apparatus is shown diagrammatically in Figure 2, and as photographed in Figure 3.

Control Panel:- The control panel, Figure 4, gives the close control of gas, air, and oxygen pressures necessary for satisfactory operation. The feed pressure manometer indicates the air pressure at the head of the feeder. The Bunsen valve, made out of a slitted piece of tubing, smooths out variations in air supply pressure. When the screw clamp is set so that about half the air entering from the air line escapes through the valve, the feed pressure manometer remains constant over considerable ranges of pressure in the air supply tank. For a large change in supply pressure, a slight change in the setting of the valve is necessary to obtain the correct feed pressure.

The burner pressure manometer is necessary to indicate the fluctuations in burner back pressure due to powder blocks which occasionally occur. A jump in burner back pressure indicates possible fouling of the product with unblown material.

The gas pressure manometer is necessary for duplication of the burner setting, in successive runs.

Connections for introducing oxygen into the air feed are provided.

Powder Feeder:- Several types of feeding apparatus were tried. Agitation in a bottle by the stream of air was only partially satisfactory since the fine material stuck to the sides of the bottle due to electrostatic effects; the collapse of bridged material caused air flow fluctuation and powder blocks in the burner; and the air

[illegible]

[illegible]

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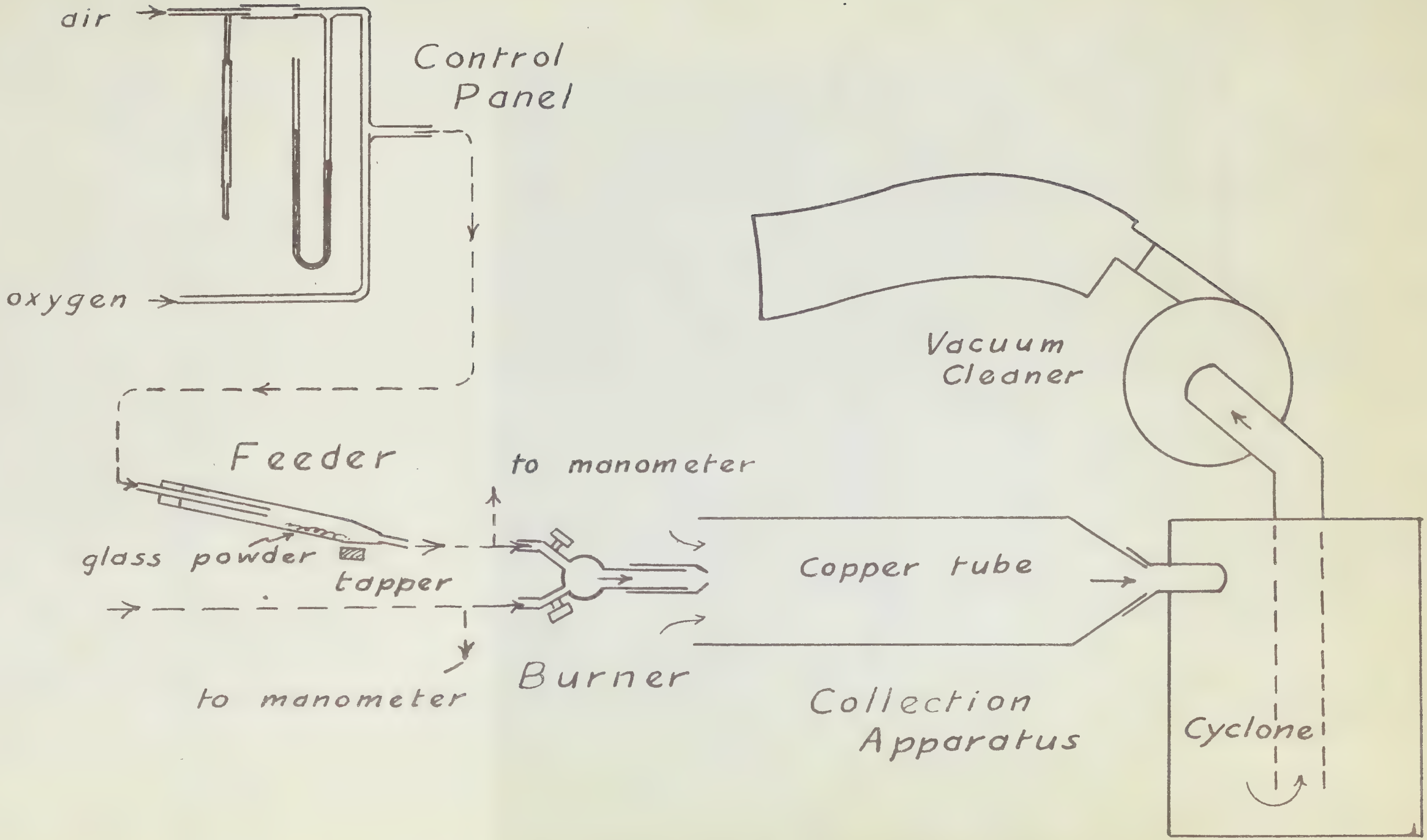


FIG. 2 - APPARATUS FOR PRODUCTION OF GLASS SPHERES

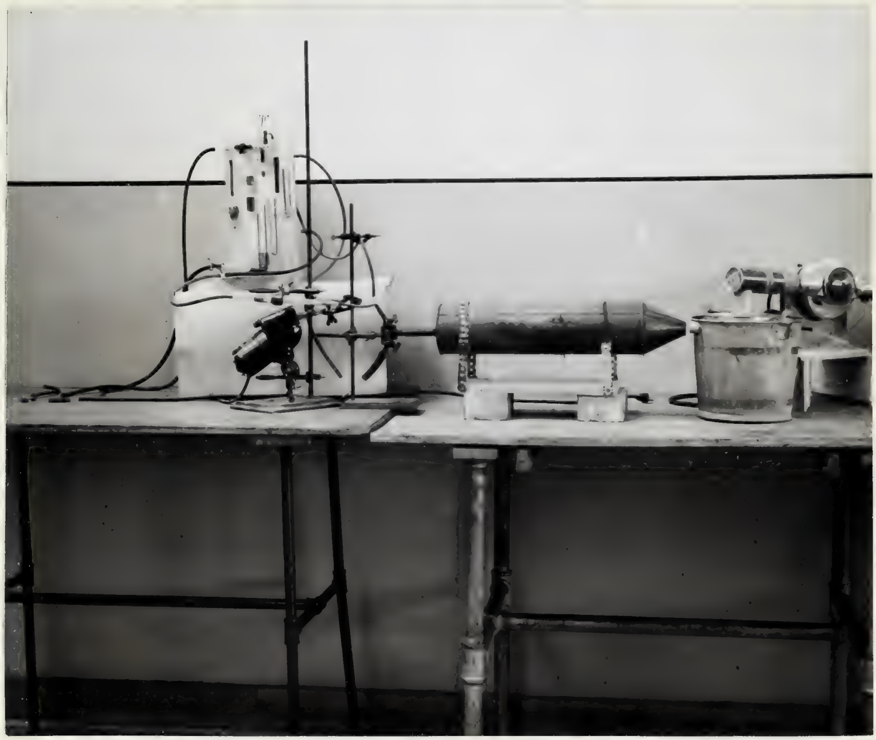
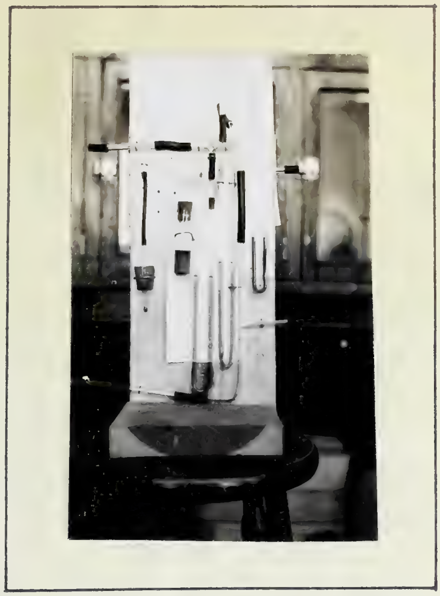


FIG. 3 - PHOTOGRAPH OF SPHERE PRODUCTION APPARATUS



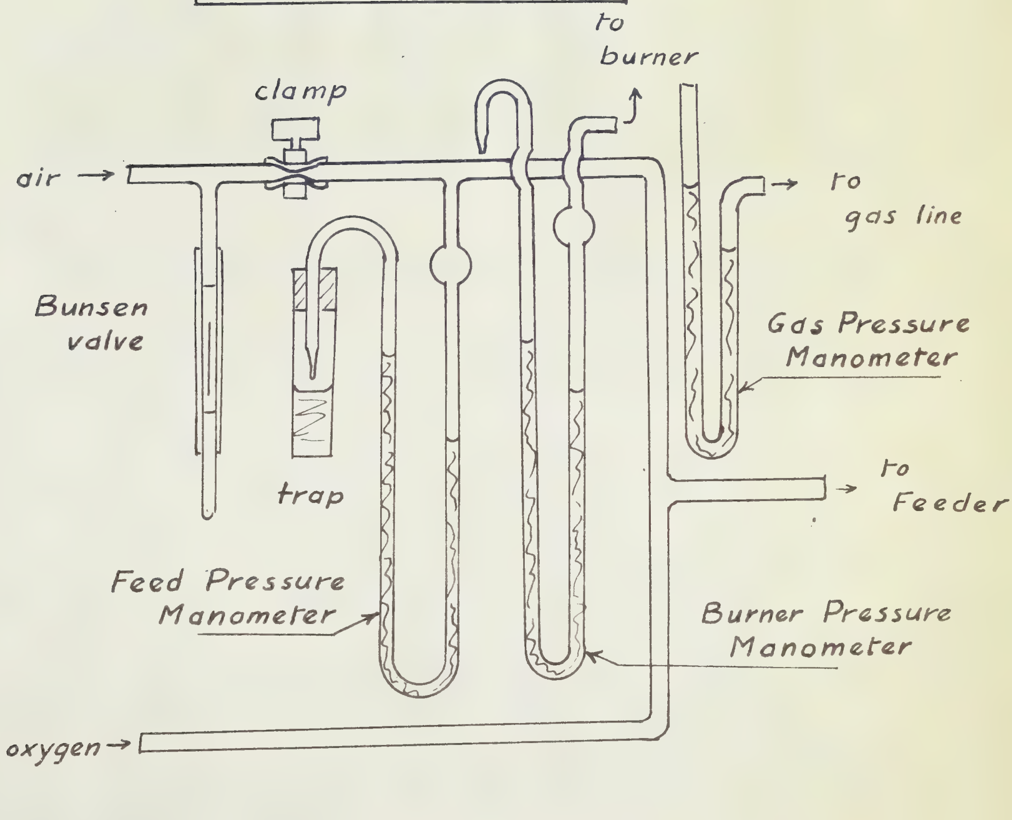


FIG. 4 - CONTROL PANEL

current would not lift the coarser material into the feeder line. To eliminate these difficulties a tube feeder with a mechanical vibrator was developed.

This feeder is shown in Figure 5 with a photograph of the actual assembly. The ground glass powder is placed in the large tube, A, which is held at an inclination of about 5 degrees by a burette clamp. The air jet tube, B, has a peripheral jet blown into the end, ~~(see detail Fig. 3b)~~, and slides through a cork, C. The jet tube is connected by rubber tubing to the air supply and the whirling jet of air, passing through the large tube, picks up the powder at the tip of the pile and carries it into the outlet and to the burner. Rate of feed is controlled manually by sliding C forward slowly as the tip of the pile of powder is blown off. A plug D fitted immediately behind the tip of the jet tube prevents the powder from eddying back into the upper end of tube, A.

The fine powder tends to stick to the inside of A due to the electric charge built up by the friction of the particle stream. This tendency is prevented by a motor driven tapper which taps the tube near the outlet. The tapper used is constructed from a 6 inch spatula held by the blade tip in a clamp, and actuated by a cam driven by a Cenco variable speed stirrer motor. The rate and intensity of the tapping can be varied, providing a second control of the feed rate.

With a little experience this feeder can be operated to give a very uniform feed rate into the burner stream even with low air velocity. It is connected to the burner by small bore rubber tubing and a rubber sleeve connection.

[illegible]

[illegible]

[illegible] envelope.

This powder is shown in figure 5 with a photograph of the feed assembly. The sample chamber powder is placed in the large tube, which is held at an inclination of about 5 degrees by a magnetic clamp. The air jet tube, a, has a [illegible] jet close into the end [illegible], and slides through a cork, c. The jet tube is connected by rubber tubing to the air supply and the whirling jet of air passing between the large tube, picks up the powder at the tip of the pile and carries it into the outlet and to the burner. [illegible]

[illegible]

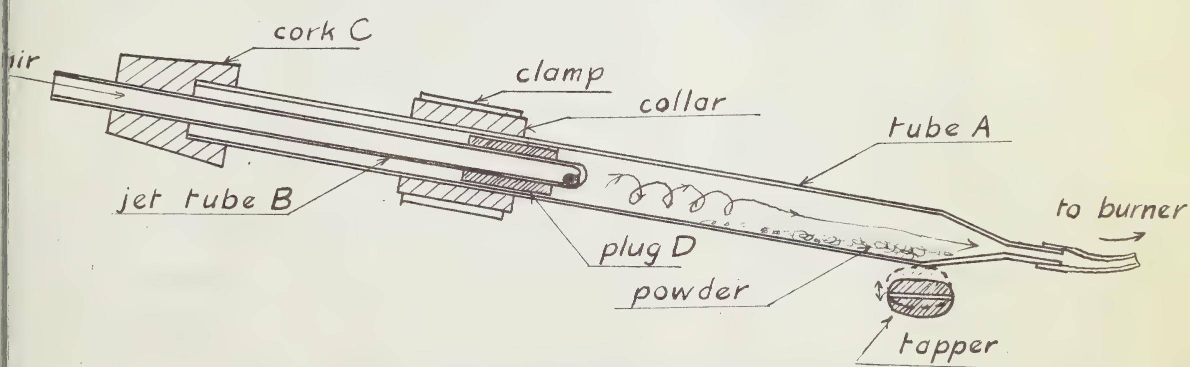
[illegible] electric [illegible] of a rotating [illegible]. This [illegible]. The [illegible] is constructed from a [illegible] in a clamp, and [illegible], producing a [illegible].

[illegible]

[illegible] velocity. It is [illegible]

[illegible]

(a) PHOTOGRAPH OF FEEDER ASSEMBLY



(b) FEEDER TUBE ASSEMBLY

FIG. 5 - POWDER FEEDER

Burner:- The burner used is an ordinary glass blowers burner fitted with a one-sixteenth inch tip. The flame found most satisfactory is an 8 inch brush flame. This retains pyrex particles, up to 30 mircons in diameter, long enough to fuse them. For pyrex spheres above 30 microns in diameter, oxygen has to be added to the air stream or a large percentage of the big particles are not fused properly. Oxygen cannot be used when feeding fine powder. The fine powder is fused into agglomerates by the high temperature, resulting in multiple spheres and more than 10 per cent. of air bubbles.

Collection Apparatus: - The collection system is composed of three parts: a 5 inch diameter copper pipe, a cyclone type collector, and a vacuum cleaner. Coarse material collects in the copper pipe. The vacuum cleaner draws the finer material over into the cyclone. Very little product reaches the vacuum cleaner bag.

The original pipe was of galvanized iron, eight inches in diameter. This was found unsatisfactory since the galvanizing flaked off and contaminated the product. Also the volume of air required to produce a carrying current for the material was too large. The five inch diameter copper pipe is satisfactory.

The cyclone collector serves two purposes. It collects almost all the product which is carried over from the copper pipe. It also cools the hot gases from the burner and thus protects the vacuum cleaner mechanism. Its construction is shown in Figure 6, and it operates as follows:

Air carrying powder enters through the inlet and follows a spiral path down the outer space. The powder particles centrifuge out and collect on the walls or fall to the bottom. The air passes out

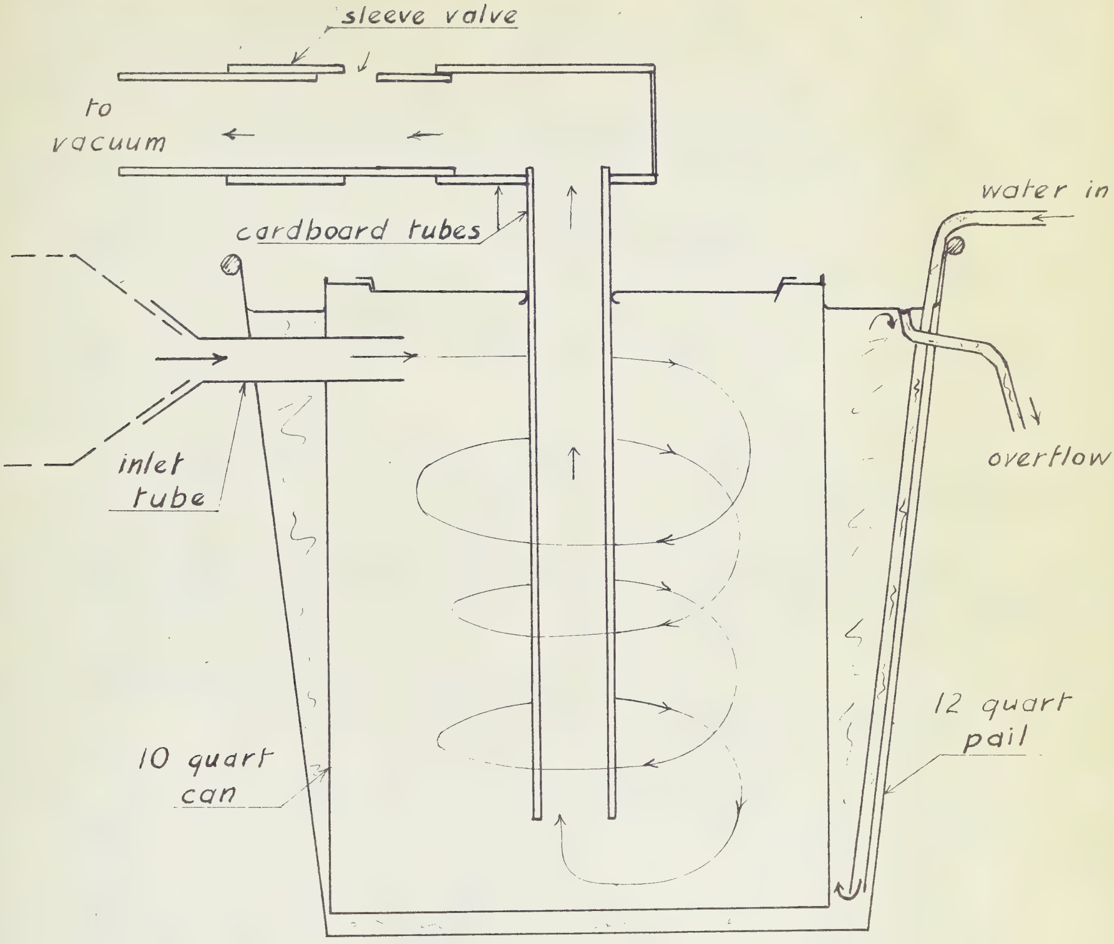


FIG. 6 - CYCLONE COLLECTOR

through the cardboard tube in the centre on its way to the vacuum cleaner.

The cyclone is water jacketed in a twelve quart galvanized iron pail. Cooling water enters the pail through a hose and overflows at the top.

The vacuum cleaner is a standard Air-Way large capacity model. The bag is the standard Air-Way paper bag, but the seam at the end is replaced by a wooden clamp to facilitate emptying.

Detailed Procedure:- Approximately 5 grams of powder are placed in the feeder tube. The jet tube is inserted and the feeder set at the correct slope.

The burner is lit and the gas adjusted to give 1 1/4 inches on the manometer. With the Bunsen valve clamp closed, the air supply valve is opened slightly. The clamp is then opened until the feed manometer indicates 1 1/4 inches. The burner pressure manometer should indicate approximately 1 inch water pressure.

The flame is checked and the vacuum cleaner is started.

The tapper is started and adjusted to give a slow steady powder feed to the burner. The burner flame should appear uniformly orange yellow, indicating uniform rate of feed and correct fusion temperature. As the powder is used up the feeder jet must be moved forward to keep the larger particles feeding. When the feeder is empty the feed connections and burner are tapped lightly to loosen adhering powder.

Control of Product:- It is advisable to clean out the cyclone between runs so that a succeeding bad run will not foul up a good

product. The product retained in the tube is not of good quality since it is above 30 microns and too large to be properly fused with air. It must be rerun later with about 1/4 inch of oxygen pressure added to the feed air pressure on the manometer reading if a plus 30 micron product is required.

It is also advisable to check each run under the microscope until operating conditions are fully standardized. The cyclone product is sampled and mounted in a drop of glycerine-water medium on a glass slide. Examination under low power magnification will indicate the quality of the product.

Results:- By use of the above apparatus and procedure, approximately 100 grams of minus 30 micron pyrex spheres were prepared. Reblowing of the tube product with oxygen, produced approximately 25 grams of minus 70 micron spheres.

The sphericity of the products exceeded 95 per cent. in all but the coarse fraction, which was approximately 90 per cent. spherical. Bubbles made up about 2 per cent. of the product but although these produced some error in elutriation they could not affect the surface measurement.

Sizing of Spheres by Elutriation

Elutriation of the glass spheres was necessary for two reasons:

(1) A series of fractions of different sizes was necessary to cover a range of specific surfaces.

(2) Microscopic measurement of particles is more accurate on sized fractions.

Apparatus:- A multi-tube elutriator of the water current type was developed which has very satisfactory operating characteristics. Figure 7 is a diagram of the apparatus as used for the sizing of glass spheres. Figure 8 is a photograph of a modified arrangement used in later experiments on pure minerals.

The apparatus may be grouped for purposes of discussion into five main components:

1. The constant pressure supply system.
2. The flow regulation apparatus.
3. The charging apparatus.
4. The elutriator tubes.
5. The flow measurement apparatus.

In addition, accessory apparatus for sample dispersion and filtration is required.

Constant Pressure Supply System: - The supply system consists of three head tanks interconnected by a siphon system, and a constant level supply tank which delivers water at constant pressure to the elutriator. This system was set up by Chalmers and Lauer during the term 1945-46 but originally had only one head tank.

Each head tank is a 40 litre carboy with a siphon outlet. Each may be filled independently from a hose line attached to a faucet, but the water must be drawn from the centre tank, which discharges by siphon through a screw clamp to the constant level tank.

The constant level tank is a 20 litre bottle equipped with an overflow tube and an outlet to the flow regulator.

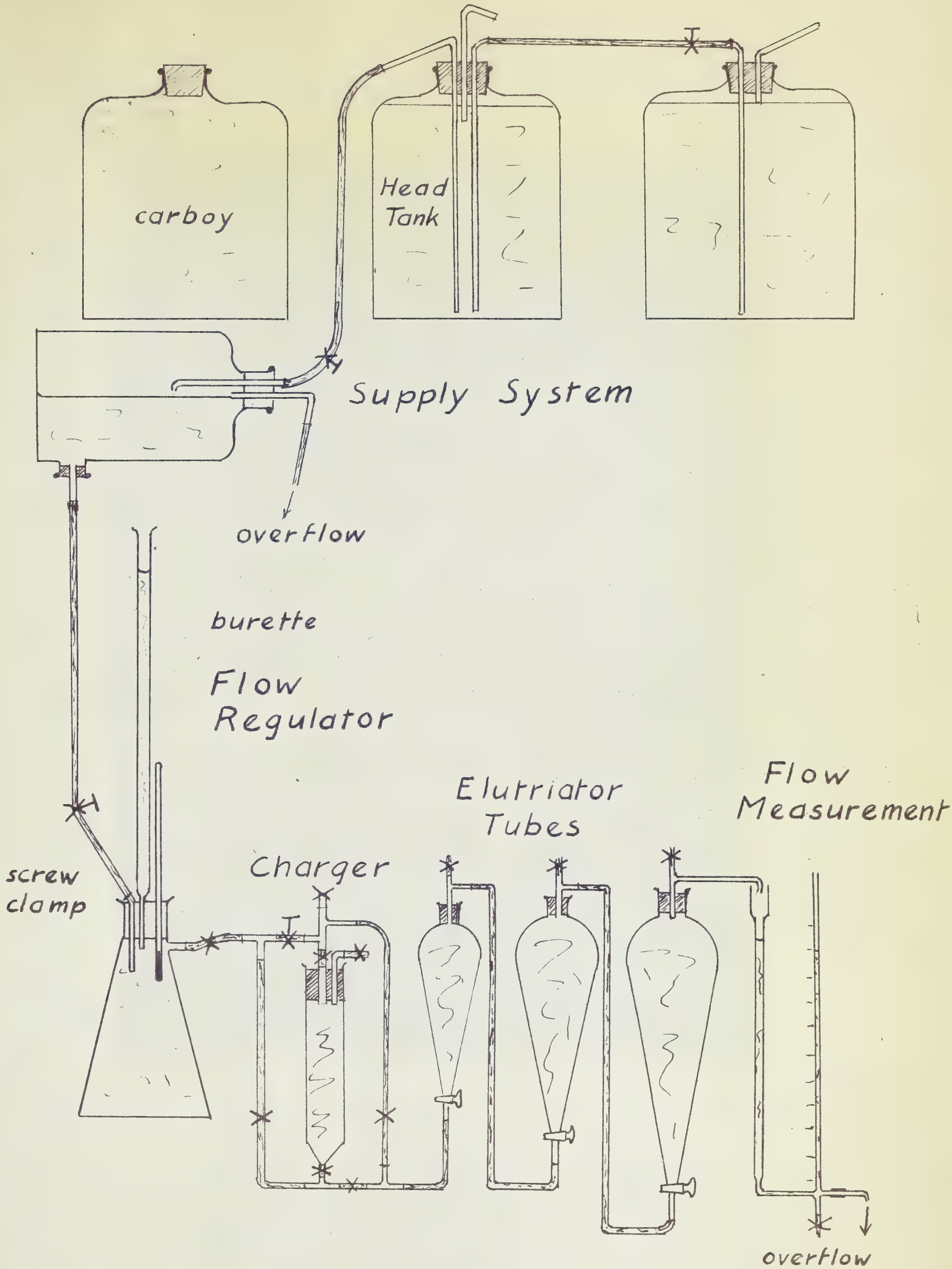


FIG. 7 - ELUTRIATOR

FIG. 8 - PHOTOGRAPH OF ELUTRIATOR

The Flow Regulation Apparatus:- The flow regulator (shown in Fig. 7) is a one litre suction flask into which is fitted a cork with inlet tube, thermometer, and Mohr burette tube. The screw clamp on the inlet line from the supply system regulates the flow to the elutriator.

The actual head of water on the elutriator tubes is indicated by the level in the burette. During a run, back pressure in the tubes falls off gradually, tending to increase the flow rate. This causes a drop in the burette, checking the increase. Flow regulation is therefore semi-automatic and during the latter part of a run no attention is required. The burette also allows escape of the air which may become entrapped in the line and gives a visual indication of the approximate flow rate.

Charging Apparatus:- Charging of the powder sample to the elutriation tubes must be done carefully to maintain free settling conditions in the tubes and to prevent air bubbles from entering.

The normal charge must be 50 grams or more to obtain fractions large enough for surface determination. Since the volume of the smallest tube is only 250 c.c. only 25 grams of fine powder can be admitted at a time. This can be regulated by the charging apparatus shown in detail in Fig. 9.

The charger tube will hold 150 c.c. of suspension. Water flow can be passed without interruption in either direction through the tube or bypassed either above or below the charger tube. The charging procedure given later illustrates the necessity of this.

Elutriator Tubes:- Sizing of the powder into fractions is carried out in the elutriator tubes of the assembly.

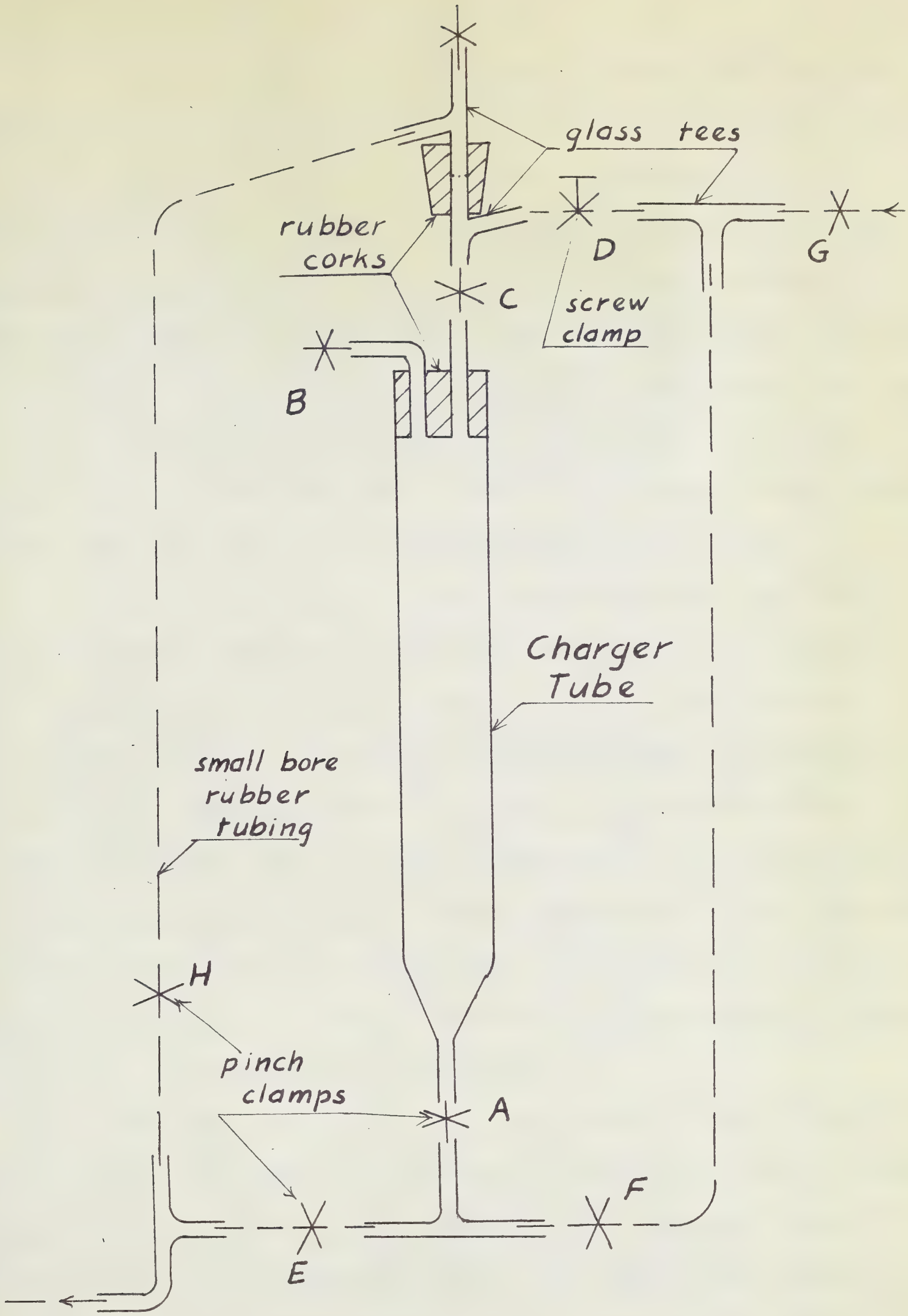


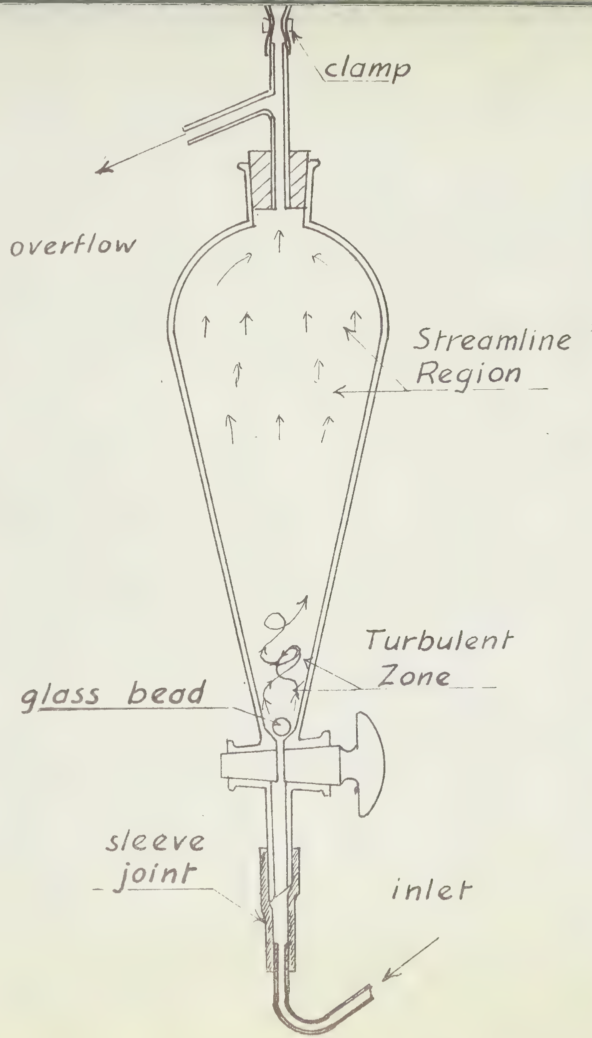
FIG. 9 - CHARGING APPARATUS

The selection of satisfactory tubes for the final design was the result of considerable preliminary testing on all available tubes.

Tests on straight tubes indicated that they were not sufficiently accurate for calibration purposes, particularly with the high velocities required to lift 30 micron spheres and with the low velocities required to retain 10 micron spheres. All fractions from 15 to 35 microns in diameter contained excessive amounts of minus 10 micron material. Observation of permanganate injected into a tube showed counter currents within the tube. Observation of powder being elutriated in the tube indicated that the velocity front was not uniform.

Tests were conducted on pear shaped separatory tubes which were found very satisfactory at all flow rates. Originally the flow was introduced through a central tube from the top. Although this arrangement performed very well when properly adjusted, difficulty was encountered in adjusting the delivery into the exact centre of the bottom of the tube. Also the exact position could not be duplicated for successive runs resulting in variable performance. It was realized that this difficulty could be eliminated by all-glass, rigid construction, but this was beyond the technical skill of the investigator so a simpler method was devised as shown in Figure 10.

The admission of flow through the bottom of the tube resulted in a jet of water which passed vertically upward in the tube and through the outlet. Therefore a device suggested by the Haultain Infrasizer was employed. A glass bead was placed in the bottom of the tube to break up the jet. Ideally the bead should seat in a ninety degree cone and this was the approximate shape of the bottom of the smaller tubes. Selection of a bead of proper size resulted in good performance in these tubes.



(a) Connections and Flow

(b) Photograph showing
Plane Velocity Front

FIG. 10 - ELUTRIATOR TUBE

Observation of permanganate and glass powder charges showed extreme turbulence at the bottom of the tube which dissipated the entrance energy so that an almost perfect plane velocity front existed in the upper half of the tube. This is shown in the photograph of a tube being charged with powder (Figure 10). The extreme turbulence was also useful in maintaining dispersion of the powder.

The inlets of the larger tubes, however, were too large and were very irregular in shape. Therefore it was necessary to place a properly shaped wax insert in the bottom as a seat for the bead, as shown in Figure 11. As this insert can be displaced by careless technique a description of the method used in its construction will be given. The tube is dried and carefully heated to slightly above the melting point of the paraffin wax. The stop cock is closed. A piece of wax slightly bigger than required is softened over a low burner flame until plastic. This is dropped into the tube and quickly pressed down with a wooden rod to seal tightly into the bottom of the tube. The tube is then heated gently until the wax in contact with the glass just melts to complete the seal. Then tube and wax are allowed to cool resulting in a wax plug (Fig. 11(a). The wooden tool (Fig. 11(b), carved to form a double edged bit as shown, is then used to drill the cone. The tool is centred at the top by a cork and is centred at the bottom by the glass tube when the cone is complete (Fig. 11(c). This ensures alignment of the axis of the cone with the tube axis. A second wooden tool (Fig. 11(d) is used to drill a 2 millimetre diameter hole from the apex of the cone to the stop-cock hole. The finished insert is shown in Figure 11(e), and provides a satisfactory seat for a 4 mm.

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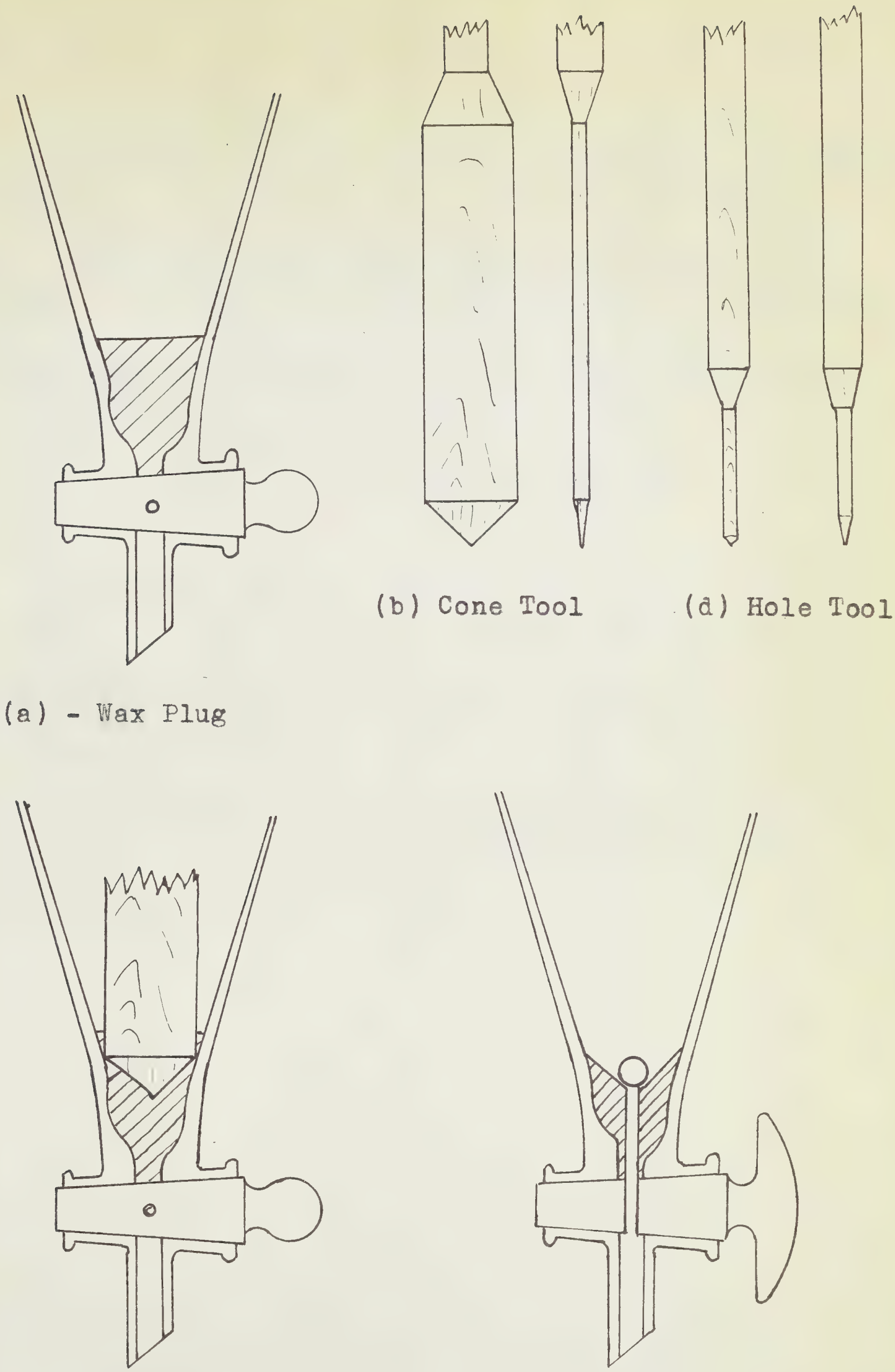
(b) Cone Tool

(d) Hole Tool

(a) - Wax Plug

(c) Drilling the Cone

(e) Completed Insert

FIG. 11 - WAX INSERT

diameter glass bead. Excess melted wax and wax turnings are removed with a cloth swab moistened with carbon tetrachloride.

Seven tubes in all were used in the investigation - two straight tubes, and five separatory tubes. The sizes of these are given in Table 1, with the theoretical minimum diameter of pyrex spheres which will settle in each tube at flow rates varying from 4 to 324 millilitres per minute, assuming streamline flow. Flow in the two small straight tubes was transitional between streamline and turbulent so values tabulated are inaccurate. Only the separatory tubes were used for glass sphere elutriation.

Connections between the tubes must be of small bore flexible rubber tubing. A larger rubber sleeve is used to join this tubing to the larger tubes as shown in Figure 10(a). Glass tubing used at the top should be of small bore. The tee and pinch cock at the top of each tube is required for release of bubbles from the system when running. The tops of all tubes must be at approximately the same height so this may be done.

Table 1. Standard Elutriator Tubes

Tube	Straight Tubes		Separatory Tubes				
	A	B	1	2	3	4	5
Volume, (ml.)			150	250	500	1000	2000
Max. Diam. (cm.)	1.15	3.4	5.4	7.0	8.5	10.0	15.0
Max. Area A (cm^2)	1.04	9.1	22.8	38.4	56.5	78.5	176.8
Flow rate Q (ml./min.)	minimum dia. of spheres in tube D (microns)						
4	31	10.5	6.5	5.1	4.2	3.6	2.9
9	46	15.7	9.9	7.6	6.2	5.4	4.3
16	62	21.0	13.2	10.2	8.4	7.2	6.9
25	76.5	26.2	16.6	12.7	10.4	8.9	7.3
36	Turbulent Flow	31.5	19.9	15.3	12.6	10.7	8.7
49		36.7	23.2	18.1	14.6	12.5	10.2
64		42.0	26.5	20.3	16.8	14.3	12.0
81		47.1	29.8	22.9	18.8	16.0	13.1
100		52.2	33.2	25.3	21.0	17.8	14.6
121		57.8	36.3	28.0	23.1	19.6	16.0
144		63.0	39.7	30.5	25.2	21.4	17.4
169		68.1	43.0	33.0	27.2	23.2	18.8
175		69.1	44.1	33.7	27.8	23.7	19.2
196		73.5	46.3	35.5	29.2	24.8	20.4
225		-	50.0	38.1	31.6	26.7	21.8
256		-	53.0	40.5	33.5	28.5	23.3
289		-	56.0	43.2	35.7	30.2	24.7
324		-	59.5	45.6	37.7	32.2	26.2

Calculated by Stokes' Law for pyrex at 20°C.

$$D = 15.8\sqrt{\frac{Q}{A}}$$

Flow Measurement Apparatus:- Accurate measurement of overflow was carried out from time to time using a graduate. However it was found that for flow regulation during charging and for indication of flow rate during the run, a flow meter was necessary. A piezometer for flow indication and measurement was constructed as shown in Figure 12(a).

Flow rate is indicated by the height of the water column necessary to cause flow through the tip. The piezometer escape tip is drawn from glass tubing and is designed to give a sensitive reading over a desired range of flow rates. Two tips were used. Calibration curves for flow rates at 20 degrees Centigrade are given in Figure 12(b).

Additional Apparatus:- (a) Dispersion apparatus, - Initial dispersion was carried out in a beaker by use of a high speed stirrer. The stirrer was an air driven rotor operating on the jet reaction principle and was patterned after a stirrer described in Industrial and Engineering Chemistry[58]. Details of construction are shown in Figure 13.

The bearings are pieces of glass tubing held in place by sections of heavy rubber tubing of correct size to fit snugly in the outer tube. These end bearing must be wired together to prevent them from being blown out. The driving rotor is blown from 5 mm. glass tubing and the impeller end is a piece of stirring rod with a rubber impeller carved from a rubber cork. The two parts are joined by a piece of rubber tubing with a hole through which air enters to the rotor. To locate the rotor in the upper bearing a thrust bearing,of two steel washers and two sections of rubber tubing,takes the outward thrust, while

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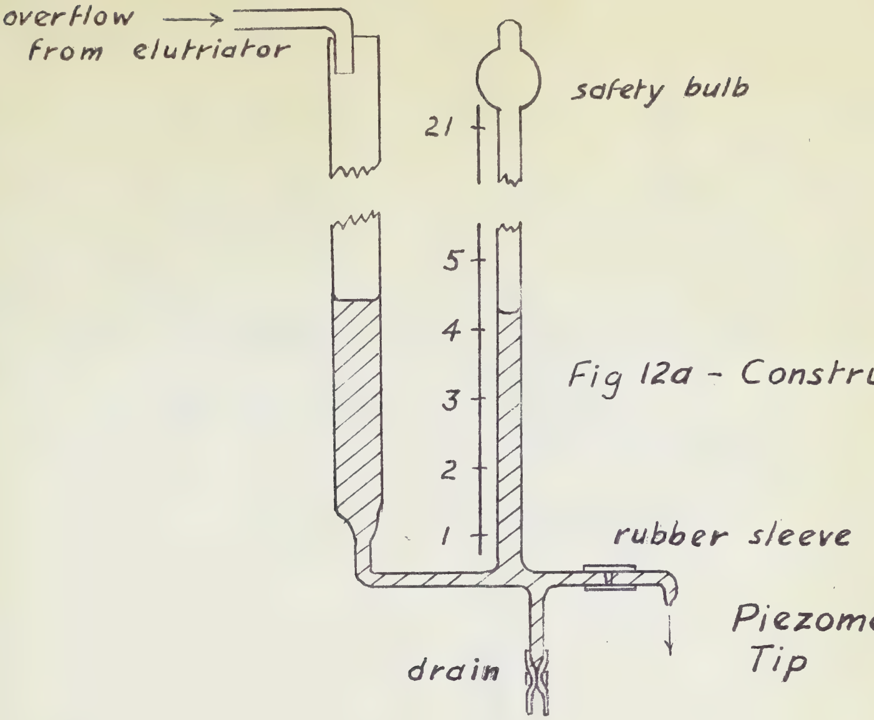


Fig 12a - Construction

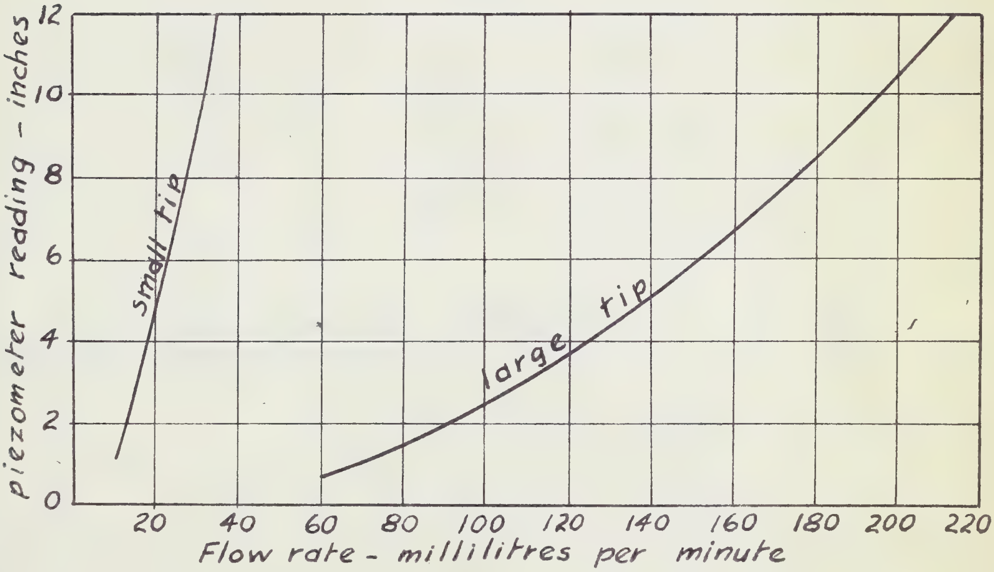


Fig. 12b - Calibration Curves at 20°C.

FIG. 12 - FLOW MEASUREMENT APPARATUS

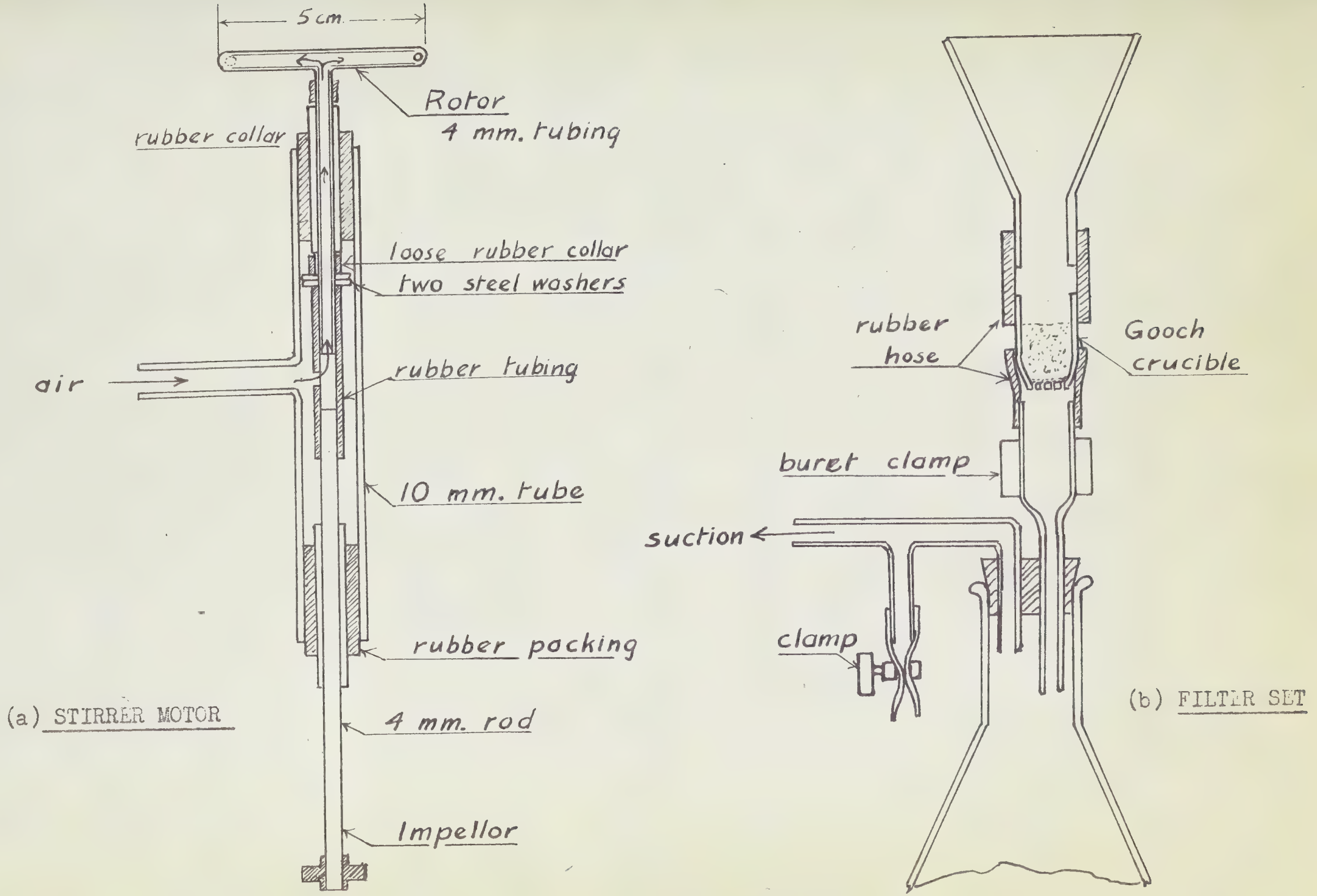


FIG. 13 - ADDITIONAL APPARATUS USED IN ELUTRIATION

a short rubber collar locates the rotor at the outer end. Oil must not be used on the bearings since a grinding compound is formed which rapidly plugs the bearings with ground glass. Five pounds of air is more than sufficient for operation.

(b) Filtration apparatus.- Filtration of the small samples is carried out on the special filter set shown in Figure 13(b).

If there is less than 10 c.c. of water to filter off or the sample is coarse, the large funnel need not be attached to the top of the crucible.

Filter discs are cut from a sheet of medium retentive filter paper. The discs are marked out with a cork borer and cut out with scissors. They should be check weighed to maintain uniformity within plus or minus 0.001 gram.

Operation Details:- Several points of procedure require explanation since they have a considerable effect upon successful operation of the process.

The water used was ordinary tap water. The head tank was filled the day before the run so that the water would be at room temperature. This prevented convection currents and ensured a constant water temperature during the run. Also dissolved oxygen, which otherwise caused small bubbles in the tubes, came out as the water warmed up to room temperature. Water stored more than a week in the warm room, became full of algal growth. It was found that about 5 c.c. of Perfex (NaOCl soln) in the tank after filling prevented this growth. There was no apparent harmful effect upon the dispersing agent during the elutriation.

Previous to the elutriation of the powder, tests to determine the optimum amount of dispersing agent were made. The method used is

described by Fryer[1] and by Bauer[59]. A series of test tubes is set up, each containing a different concentration of the dispersing agent. A small sample of the powder is placed in each tube and all are agitated at the same time. The tubes are then left standing for an hour and examined. It will be seen that too little or too much dispersion causes flocculation. The optimum amount can be selected by comparing the turbidity in the tubes.

Calgon was used as a dispering agent. The optimum amount for pyrex spheres was determined as 0.2 grams per litre. This was used for initial dispersion and also was added to the water in the head tank just before a run to maintain dispersion. It was found that the sodium phosphate greatly accelerated the growth of algae so water containing Calgon could not be stored for more than a day.

Since the viscosity of water varies considerably with temperature, the nominal rate of flow for 20 deg. Centigrade (from Table 1) must be corrected for other temperatures. Correction factors for temperatures between 15 deg. and 25 deg. C., taking changes in viscosity and specific density into account are given in Table 2. The factors were calculated from the formula:

$$f = \frac{(d_1 - d_2)_T}{(d_1 - d_2)_{20^\circ}} \times \frac{U_{20^\circ}}{U_T}$$

where d_1 = density of pyrex (gm. per c.c.)

d_2 = density of water (gm. per c.c.)

U = viscosity (poises)

f = factor for flow rate correction.

Table 2. Temperature Correction for Elutriator Flow Rate.

(1) Temp. degrees C.	(2) Viscosity U (poises)	(3) Viscosity Correction U_{20}/UT	(4) Density d_2 gms. per cc	(5) Density Correction $\frac{(d_1-d_2)T}{(d_1-d_2)_{20}}$	(6) Correction Factor f (3) x (5)
15	1.1404	.882	0.99912	0.9997	0.881
16	1.1111	.905	0.99897		0.904
17	1.0828	.928	80		0.927
18	1.0559	.952	62		0.952
19	1.0299	.976	43		0.976
20	1.0050	1.000	23	1.0000	1.000
21	0.9810	1.024	02		1.024
22	0.9575	1.050	0.99780		1.050
23	0.9358	1.074	56		1.074
24	0.9142	1.101	32		1.102
25	0.8937	1.125	0.99707	1.0010	1.126

To obtain actual rate, multiply flow rate at 20°C. by "f" for actual temperature.

Outline of Procedure:- For elutriation of glass spheres only four tubes were available, the 150, 250, 500 and 1000 millilitre separatory tubes. For reference these were designated Tubes 1, 2, 3 and 4, respectively.

From Table 1 flow rates and tube sizes were selected to give a set of powder fractions whose minimum diameters were approximately a geometric series. With the tubes available, in order to cover a suitable range, the procedure was divided into three stages. Figure 14 shows the three flow rates chosen and the tubes used with each flow rate.

Unsized Sample

STAGE I

Flow rate 81 ml. per min.

Tube 3

Coarse Portion

+18.8 microns

STAGE II

Flow: 175 ml. per min.

Tube	Size (Nominal) microns	Fraction Number
1	+ 44.1	1
2	-44.1 + 33.7	2
4	-33.7 + 23.7	3
o'flo	-23.7 + 18.8	4

Fine Portion

-18.8 microns

STAGE III

Flow: 16 ml. per min.

Tube	Size (nominal	Fraction Number
1	-18.8 + 13.2	5
4	-13.2 + 7.2	6
o'flo	-7.2	7

Fig. 14 Procedure for Elutriation of Microscopic Spheres

In Stage I the sample is split into two fractions, one nominally coarser than 18.8 microns in diameter, the other finer. This is accomplished by elutriating the sample for two hours with a flow rate of 81 millilitres per minute in Tube 3. In Stage II the coarser portion is re-run at 175 millilitres per minute using Tubes 1, 3, and 4 to obtain four coarse fractions. In Stage III the fine portion from Stage I is re-run at 16 millilitres per minute, using Tubes 1 and 4 only,

to obtain two fine fractions and an overflow product. It will be noticed that had Tube 5 been available for glass sphere elutriation, a better selection of rates could have been made requiring only two stages. (see Table 1)

Detailed Procedure: Stage I

Apparatus: Tube 3 only

Preliminary Steps: (a) Fill head tank the day before.

(b) Add 0.2 grams Calgon to head tank for each litre of contents.

(c) Couple up elutriator, having current by-passing above the charger tube, clamps G, D, and H open, all others closed (see Figure 9).

(d) Allow tube to fill and eliminate all air.

(e) Adjust screw clamp to regulate flow to slightly less than 81 ml. per min. as indicated by piezometer (Figure 12).

(f) Adjust flow into the constant level tank so that it is just overflowing.

(g) Note temperature and calculate flow rate equivalent to 81 ml. per min. at 20 degrees C. (see Table II).

Dispersal of Sample: (a) Place sample in 200 ml. beaker.

(b) Add 100 c.c. water and 0.2 gm. Calgon.

(c) Stir rapidly for 5 minutes with air stirrer.

(d) Raise stirrer and wash off impeller with wash bottle.

Charging: (a) Remove charger tube and cork from apparatus with clamp A in place.

(b) Wash sample into charger.

(c) Place cork in tube, clamp B open.

(d) Connect charger into apparatus.

(e) Divert water flow to by-pass below the charger by opening clamps E and F and closing D.

(f) Open clamp A and let charger tube fill slowly from the bottom, air escaping through B.

(g) Open Clamp C momentarily to clear outlet tube of air bubbles.

(h) When tube is full and all air has escaped, close B, close A, and open C.

(i) Quickly close E and open A. Charge should begin to lift and charge out C into Tube 3.

(j) Open D slightly until heavy material just teeters at A. This bypasses water above the charger and keeps charge in Tube 3 at low density.

(k) Regulate flow rate to just below the required rate.

(l) As finer material becomes charged, close D and let coarser material lift into Tube 3.

Running: (a) Run for 2 hours after Tube 3 overflows.

(b) As soon as the material begins to overflow Tube 3, regulate flow to the correct rate and check with graduate. Note exact piezometer setting and regulator burette reading. Reset head tank flow if necessary.

(c) Rate of flow requires manual regulation during the first half hour, but is semi-automatic for the remainder of the run.

(d) Collect overflow in 10 quart enamel pails.

Draining Tubes: At end of run, quickly shut off Tube 3 and stop cock A.

(b) Shut off regulator clamp and head tank.

(c) Drain tube and charger into same beaker. To drain the tube, remove rubber tubing from the bottom and wash tubing into beaker. Then open stop cock on the tube and open pinch cock at the top. Insert a

piece of fine wire up into the tube to lift the bead. When tube has drained, wash down with wash bottle.

Stage II

Apparatus:- Tubes 1, 2 and 4, connected as in Figure 1.

Preliminary Steps:- as for Stage I.

Dispersal of Sample:- (a) Allow coarse fraction from Stage I to settle and siphon off water except for about 100 millilitres.

(b) Disperse as for Stage I, for 2 minutes only.

Charging:- (a) to (l) Repeat as for Stage I.

In some cases the bulk of water in a rerun sample cannot be kept to one charger load. In these cases the sample may be charged in two or three loads, if the charger is completely cleared each time. The following additional charging steps are necessary.

(m) To finally transfer the coarsest material into Tube 1, open E and close H, and after nearly all the charge has settled and passed out through E, open D, close F and drive the remainder into tube 1.

(n) When charge is all in, open F, close D, C and A.

(o) Charger may then be removed, refilled, and steps (a) to (n) repeated.

Running:- As for Stage I.

Tap side of Tube 4 gently every 20 minutes to dislodge fines.

Draining:- As for Stage I.

Drain tubes in numerical order into marked beakers.

Filtration of Fractions:- (a) Let each fraction stand until clear (overnight for fine fractions).

(b) Siphon off most of the water.

(c) Filter into previously weighed gooch crucible (see Fig. 13(b).

(d) Change receiver flask and wash with 3 ml. 'used' alcohol to remove water and prevent caking.

(e) Wash with 1 to 2 millilitres clean alcohol (retain washings as 'used' alcohol).

Drying of Fractions:- Dry sample thoroughly at low heat over a hot plate and cool in a dessicator.

Weighing:- Weigh to nearest 0.005 gm.

Stage III

Apparatus:- Tubes 1 and 4 only.

Preliminary:- As for Stage I.

Sample Dispersal:- Overflow collected in Stage I, settled 4 hours and water siphoned off.

Disperse as for Stage I.

Charging:- As for Stage II. Flow rate under 16 millilitre per minute.

Running:- As for Stage II but run for 3 hours after material overflows tube 4, at 16 ml. per minute.

From time to time agitate the rubber tubing connecting the tubes to dislodge the fine material which tends to settle out of the slow current at the bottom of the tubing loops.

Draining, Filtering and Drying:- As for Stage II.

Results: Each size fraction as obtained from elutriation was placed in the same bottle as previous fractions of the same size. The total quantity of each fraction is given in Table 3.

Table 3 Results of Sphere Elutriation

Fraction	Nominal Size (microns)	Total Weight (grams)
1	+ 44.1	6.2
2	+ 33.7	12.3
3	+ 23.7	38.4
4	+ 18.8	22.4
5	+ 13.2	30.3
6	+ 7.2	13.9
7	- 7.2	0.5

Each fraction was sampled and size analysed by microscope. Final results are given in Tables 5, 6 and 7 and are shown graphically in Figure 19.

Microscopic Evaluation of Specific Surface

The surface of each fraction was determined accurately by a Microscopic Method, involving the preparation of mounts, making of photo-micrographs, measurement and count of particles, and calculation of specific surface.

Preparation of Mount: Preparation of a suitable mount for photo-micrography involves the technique of sampling, dispersion and mounting discussed in the theoretical section.

Sampling: A small sample must be taken accurately. The bottle containing the fraction is shaken for five minutes to thoroughly mix the powder. The sample is then poured out on a clean sheet of hard white paper and coned and quartered to reduce it to about 0.5 grams. From this is taken a 0.01 gram sample.

Dispersion: The sample is placed in a small mixing cell, which is the glass base from a burnt out fuse. The dispersion medium used is a five to one glycerol-water solution. This has a refractive index of about 1.43 as compared to pyrex with a refractive index of 1.46, and is viscous enough to hold the powder in suspension for some time. Sufficient medium is added to the sample in the cell to give the required density of suspension. The powder is mixed thoroughly with the medium using a small glass rod tipped with a short section of small bore flexible rubber tubing.

Mounting: When the suspension is uniform a drop is transferred quickly to a glass slide and covered quickly with a cover glass. The cover glass is pressed down and rotated gently to spread the glycerine-powder mix to a layer one particle deep.

Preliminary investigation under a medium power microscope is advisable to check uniformity and density of the mount. The slide should show no flocculation, should be perfectly uniform in density and should be dense enough to give a good field for counting. If too dense add more medium to the mixing cell and make another mount. It is important that the difference in refractive index between the pyrex and the medium should give low relief. However if relief is too low for good definition of particles, a little water should be added to the medium.

Photomicrography: Photomicrographs were made on 8 by 10 inch Photostat paper at 258 X magnification using the optical equipment shown in Figure 15.

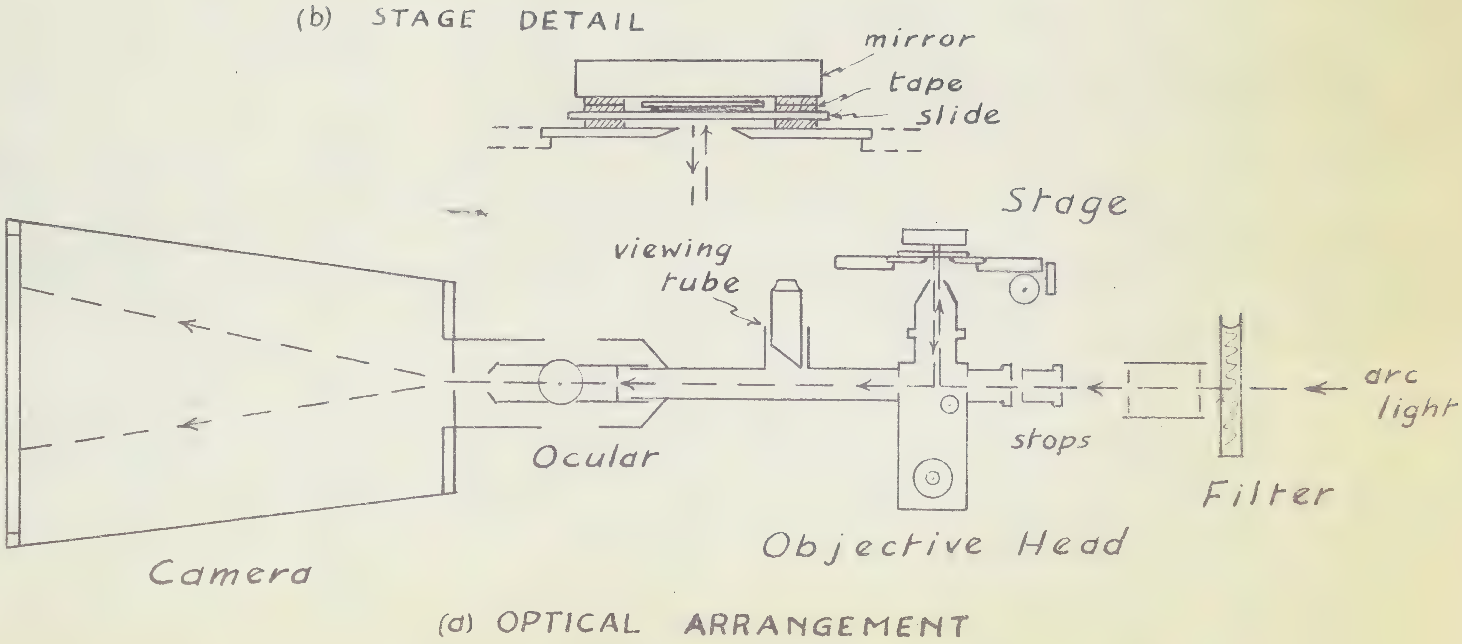


FIG. 15 - APPARATUS USED FOR PHOTOMICROGRAPHY

Optical Arrangement: Light from the arc lamp passes through the water filter, filter disc, and diaphragm stops, into the objective head, where it is reflected up through the objective lens to the mount. The mount is placed upon the stage with a plane mirror above as shown in the detail (Fig. 15b). To prevent the slide from being moved by vibration it is supported on two strips of friction tape on the stage. The mirror is spaced away from the slide by two other strips of tape.

The light is reflected back down through the mount and passes into the camera through the ocular and shutter.

Calibration of the Ocular Scale: The regular ocular is replaced by a Spender 10X filar ocular. This is adjusted to focus the ocular scale on the camera ground glass. Calibration of the projected scale is carried out against a 0.01 millimeter mirror stage micrometer. The micrometer is placed face down on a glass slide plate lying on the stage. The objective is focussed on the micrometer scale and the image is projected on the camera ground glass. Calibration is carried out by direct comparison of the projected ocular and micrometer scales.

With a 16 millimetre objective and the camera extension at 55, one small division of the filar scale equals 13.7 microns, giving a magnification of 258 X.

Procedure: (1) Optical adjustments:

(a) Filter - green liquid.

(b) First stop - 3.5

(c) Second stop - 3/4

(d) Objective - 16 mm.

(e) Spencer filar ocular.

(f) Camera Extension 55.

(2) Focussing:

(a) Place slide on stage with mirror on top, not touching the cover glass.

(b) Check focus of filar scale on ground glass.

(c) Using auxiliary focussing tube, select a field at random. Focus on first image, which is in the same plane as the stage micrometer. (Second image is a mirror reflection.)

(d) Project on camera ground glass and focus sharply.

(3) Photography:

(a) Expose photostat paper 10 seconds.

(4) Develop 45 seconds in News Bromide PMC Developer.

(5) Fix and wash.

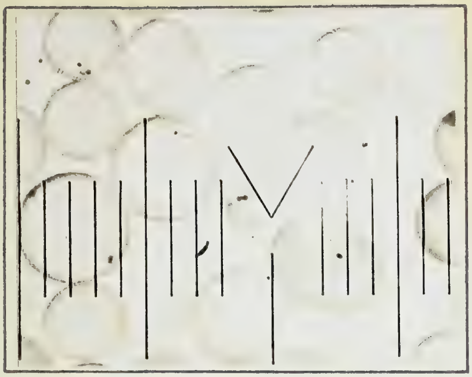
Results: A sample of the paper negative obtained is shown in Figure 16.

Two fields selected at random were photographed from each mount. Two mounts were prepared from each of fractions 3, 4, and 6, and one from each of fractions 1, 2 and 5. No significant difference in size distribution was encountered between two mounts from the same fraction. (See results of measurement and count, Tables 5, 6, 7).

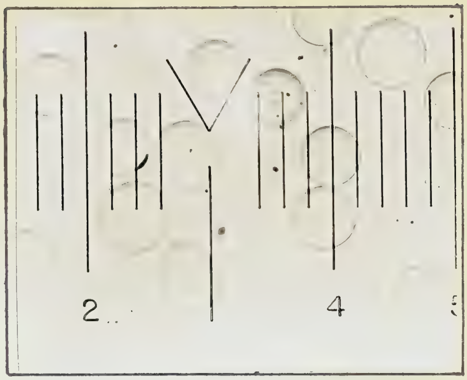
Since counting and measuring of the particles could be done from the negatives, prints were unnecessary. However, to illustrate the size of each fraction, small prints were made, as shown in Figure 17.

Since these prints cover only a small portion of the field they are useless for quantitative size analysis. They do show the range

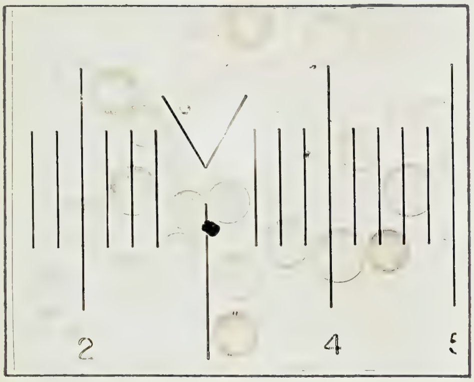
FIG. 16 - SAMPLE OF PHOTOMICROGRAPHY NEGATIVE



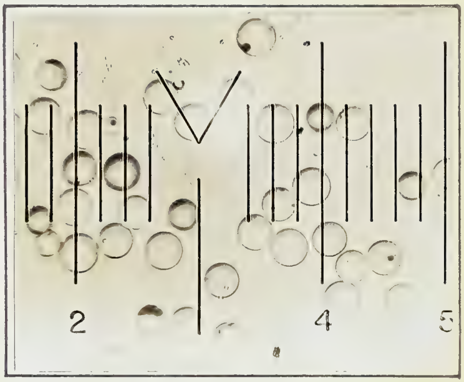
Fraction 1
D_s 50.0 microns



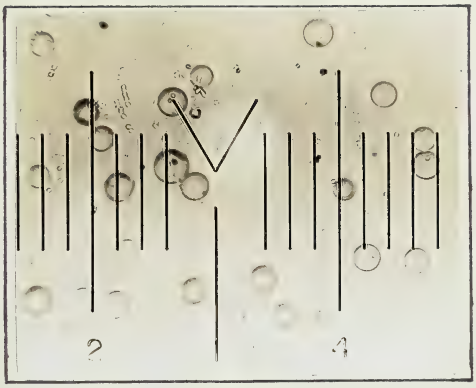
Fraction 2
D_s 35.9 microns



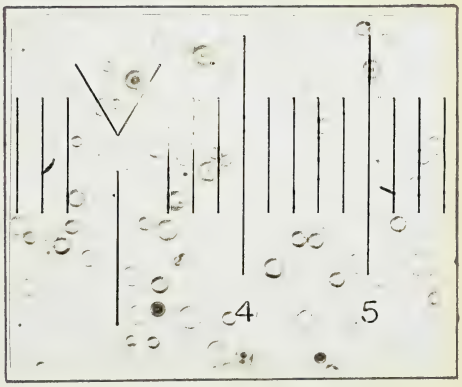
Fraction 3
D_s 27.0 microns



Fraction 4
D_s 20.8 microns



Fraction 5
D_s 15.8 microns



Fraction 6
D_s 10.1 microns

1 scale division = 13.7 microns

FIG. 17 - PHOTOMICROGRAPHS OF SPHERICAL FRACTIONS

of sizes in each fraction, the sphericity of the particles, and the definition obtained in the photomicrographs. Apparently opaque spheres are bubbles.

Measurement and Calculations: Measurement and count of the spherical particles was carried out on the paper negative using a plastic scale.

Scale: The scale of the filar micrometer as projected on each picture was useful for comparative purposes. However it could not be moved around on the paper. Measurement with dividers and comparison with the scale proved tedious and involved interpolation between scale divisions. The plastic measuring scale shown in Figure 18 was devised and was found to be accurate and rapid.

The scale is constructed as follows: From the calibration of the ocular system, 5 scale divisions equal 68.5 microns. On a piece of plastic sheet a horizontal base line is scribed and marked off into more than 68.5 equal divisions as shown in the figure. These divisions may be any length depending on the accuracy required in the instrument. At the 68.5 mark a short vertical line, exactly equal to 5 photographic scale divisions, is scribed. A third line is scribed through the zero of the base line and the upper end of the vertical line. To make the lines and scale more legible the scribed marks are filled with red grease pencil.

It is evident that the diameter of any sphere fitting between the two lines will be given very nearly by the scale reading where the sphere is tangent to the base line. The theoretical error in reading the diameter on the base line can be shown to be 0.5 per cent. at this

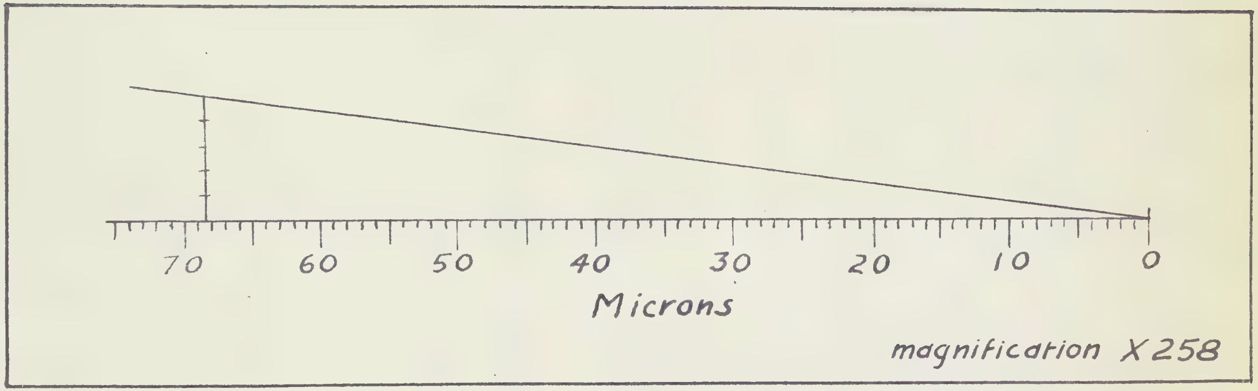


FIG. 18 - SCALE FOR MEASUREMENT OF PARTICLES

slope or 0.1 micron for a 20 micron sphere. Increasing the length of the base line and decreasing the slope would improve the accuracy.

The instrument may be manipulated with one hand while measurements are tabulated with the other.

Method: Using the above instrument all the particles on a negative are measured to the nearest micron. Since the images of particles around the edge of the plate are elongated radially from the optic centre the base line should be kept pointing toward the centre of the plate. The diameters can be judged within 0.5 microns quite easily if the particles are in focus. Sub-angular particles, being few in number, do not cause an appreciable error if their diameters are approximated.

Tabulation: The measurements are tabulated as shown in Table 4, which also shows a sample calculation for average diameter and specific surface.

Results: The results of the microscopic count and the calculations of specific surface on the spherical pyrex powder fractions are given in Tables 5, 6 and 7.

Table 4 - Specimen of Tabulation, and Calculation of Microscopic Results

Fraction number: x

Plate number: xx

Measured Values			Calculated Values		
Diameter D (microns)	Count	Total n	%	nD^2	nD^3
12		-	-	-	-
13	/	1	2.8	169	2200
14	//	2	5.6	392	5490
15	卌	5	13.9	1125	16900
16	卌 ///	8	22.2	2040	32800
17	卌 卌	10	27.8	2890	49100
18	卌 /	6	16.7	1950	35000
19	///	3	8.3	1083	20600
20	/	1	2.8	400	8000
21		-	-	-	-
		36	100	10,050	170,100

Average Diam. $D_s = \frac{nD^3}{nD^2} = \frac{170{,}100}{10{,}500} = 16.9$ microns

Specific Surface $S = \frac{60{,}000}{d_1 D_s} = \frac{60000}{2.23 \times 16.9}$

$= 1580$ sq. cm. per gm.

Table 5 - Results of Microscopic Size Analysis of Spherical Fractions 1 and 2

Fraction 1			Fraction 2		
Measured Diam. (microns)	Count	%	Measured Diam. (microns)	Count	%
29-30	3	1.6	10	1	0.5
31-32	1	0.6	13	3	1.5
33-34	2	1.1	15	1	0.5
35-36	0	-	17	2	1.0
37-38	2	1.1	20	1	0.5
39-40	4	2.2	28	1	0.5
41-42	11	6.1	29	2	1.0
43-44	20	11.1	30	4	2.0
45-46	41	22.6	31	9	4.4
47-48	31	17.2	32	16	7.8
49-50	20	11.1	33	24	11.8
51-52	7	3.9	34	25	12.3
53-54	11	6.1	35	26	12.8
55-56	10	5.5	36	23	11.3
57-58	5	2.7	37	21	10.3
59-60	5	2.7	38	11	5.4
61-62	2	1.1	39	9	4.4
63-64	3	1.6	40	13	6.4
65-66	3	1.6	41	4	2.0
67-68	0	-	42	4	2.0
69-70	1	.6	43	1	0.5
	N = 181		44	1	0.5
			45	1	0.5

Fraction 1:

$nd^2 = 436{,}800$

$nd^3 = 21{,}962{,}000$

$D_s = 50.0$

$S = \dfrac{60000}{50 \times 2.23} = 540$

Nominal +43 - 73

Fraction 2:

N = 203

$nd^2 = 247{,}900$

$D_s = 35.9$

$S = 750$

$nd^3 = 8{,}882{,}000$

Table 6 - Results of Microscopic Size Analysis of Spherical Fractions 3 and 4

	Fraction 3			Fraction 4		
(microns)	Mount 1	Mount 2	Combined	Mount 1	Mount 2	Combined
10				0.6	0.1	0.3
11				0.6	0.1	0.3
12				0.2	0.6	0.4
13				0.7	1.2	1.0
14	0.3	0.6	0.5	0.2	1.5	0.9
15		0.3	0.2	0.9	1.0	1.0
16	0.55		0.2	1.9	2.8	2.4
17	0.55	0.3	0.5	4.3	7.1	5.8
18				9.3	10.0	9.6
19	0.55		0.3	10.5	9.8	10.0
20	0.55	1.1	0.8	21.8	19.6	20.5
21	1.6	1.4	1.5	22.0	19.3	20.5
22	2.2	3.3	2.7	11.7	16.8	14.5
23	6.5	5.5	6.1	8.5	6.5	7.4
24	10.4	13.1	11.5	3.9	1.8	2.7
25	14.2	17.5	15.6	1.9	1.0	1.4
26	10.4	15.3	12.5	0.2	0.4	0.3
27	10.6	11.6	11.1		0.3	0.2
28	8.2	9.1	8.6	0.6		0.3
29	9.0	6.2	7.8	0.2		0.1
30	11.4	7.3	9.8			
31	5.2	3.3	4.4	0.4		0.2
32	3.3	1.8	2.7			
33	2.5	1.4	2.0			
34	1.1	0.6	0.8			
35	0.8	0.6	0.6			
N	367	275	642	538	674	1212
D_s	26.9	27.1	27.0	21.2	20.4	20.8
S	1010	990	1000	1270	1310	1290

Table 7 - Results of Microscopic Size Analysis of Spherical Fractions 5 and 6

Size Microns	Fraction 5 Mount 1 %	Fraction 5 Mount 2 %	Fraction 5 Combined %	Fraction 6 Mount 1 Field 1	Fraction 6 Mount 1 Field 2	Fraction 6 Mount 2	Fraction 6 Combined
3				0.4	0.4		0.23
4				0.8	0.9	0.3	0.6
5				1.1	0.4	1.2	0.9
6				5.3	3.5	3.6	4.1
7				11.4	7.4	9.2	9.5
8	.3		.2	17.4	13.0	12.0	14.1
9	1.2		.6	18.5	19.2	18.6	19.0
10	1.2	2.0	1.5	21.9	26.9	24.7	24.7
11	3.2	2.0	2.6	11.7	17.4	13.8	14.3
12	6.1	4.9	5.6	4.2	7.8	10.4	8.0
13	9.0	10.5	9.7	4.9	1.7	2.6	3.1
14	14.8	12.8	13.9	2.3	1.3	1.2	1.5
15	22.3	20.1	21.4			0.3	0.1
16	15.4	16.4	15.7				
17	16.9	14.1	15.5				
18	6.4	12.2	10.7				
19	1.4	3.3	2.3				
20	1.4	2.3	1.9				
N	343	304	647	264	230	382	876
D_s	15.6	16	15.8	10.0	10.1	10.2	10.1
S	1740	1680	1710	2680	2660	2640	2660

Discussion of Results

A summary of the surface standards which were prepared is given in Table 8.

Table 8 - Standard Surface Fractions

Pyrex Fraction	Nominal Size (microns)	Measured Average Diameter D_s (microns)	Specific Surface S cm^2 per gram
1	+ 44.1	50.0	540
2	-44.1 + 33.7	35.9	750
3	-33.7 + 23.7	27.0	1000
4	-23.7 + 18.8	20.8	1290
5	-18.8 + 13.2	15.8	1710
6	-13.2 + 7.2	10.1	2660

The range of particle sizes covered by the six powder fractions extends from 50.0 microns (equivalent to Tyler 280 mesh) down to material averaging about 10 microns in diameter (theoretical 1500 mesh). These standards therefore cover the range of particle sizes considered most important in ore dressing operations.

Accuracy of the standards can be judged by inspection of Tables 6 and 7. The results on two mounts of Fraction 3 deviate by only 1 per cent. from the mean, which would indicate a difference in average diameter of about 0.1 micron due either to measurement or sampling errors. The deviation is 1 1/2 per cent. for Fraction 4 and is 1 3/4 per cent. for Fraction 5. Results for Fraction 6 show the least deviation, slightly less than 1 per cent. The good agreement between the two fields, shown for Fraction 6, is indicative of the good dispersion and uniformity of mount obtainable with very fine powders.

Table 5 shows only the total counts for Fractions 1 and 2 since it was necessary to combine all results in each case to get a large enough count. The large particles are relatively the most accurately measured but error may be due to an insufficient number of particles. However, from Tables 6 and 7 it would seem that with a count of about 200, results should deviate from the mean by less than 2 per cent., so the error in Fractions 1 and 2 is probably not more than this.

Figure 19 is a plot of the size distribution of the six fractions. It is evident that the size range for each fraction is within 15 microns except for a small percentage, the surface of which, if neglected, would not make a significant difference. Therefore no error in results should be expected due to insufficient depth of focus.

The sample photomicrograph, Figure 16, shows that the percentage of sub-angular particles can make no significant error in results. The bubbles are all closed and were measured as solid spheres so they can cause only an insignificant error in results, due to error in material density.

The specific surface of the standards powders can therefore be considered as being accurate within 2 per cent., which is well within the reproducibility of the air permeability apparatus.

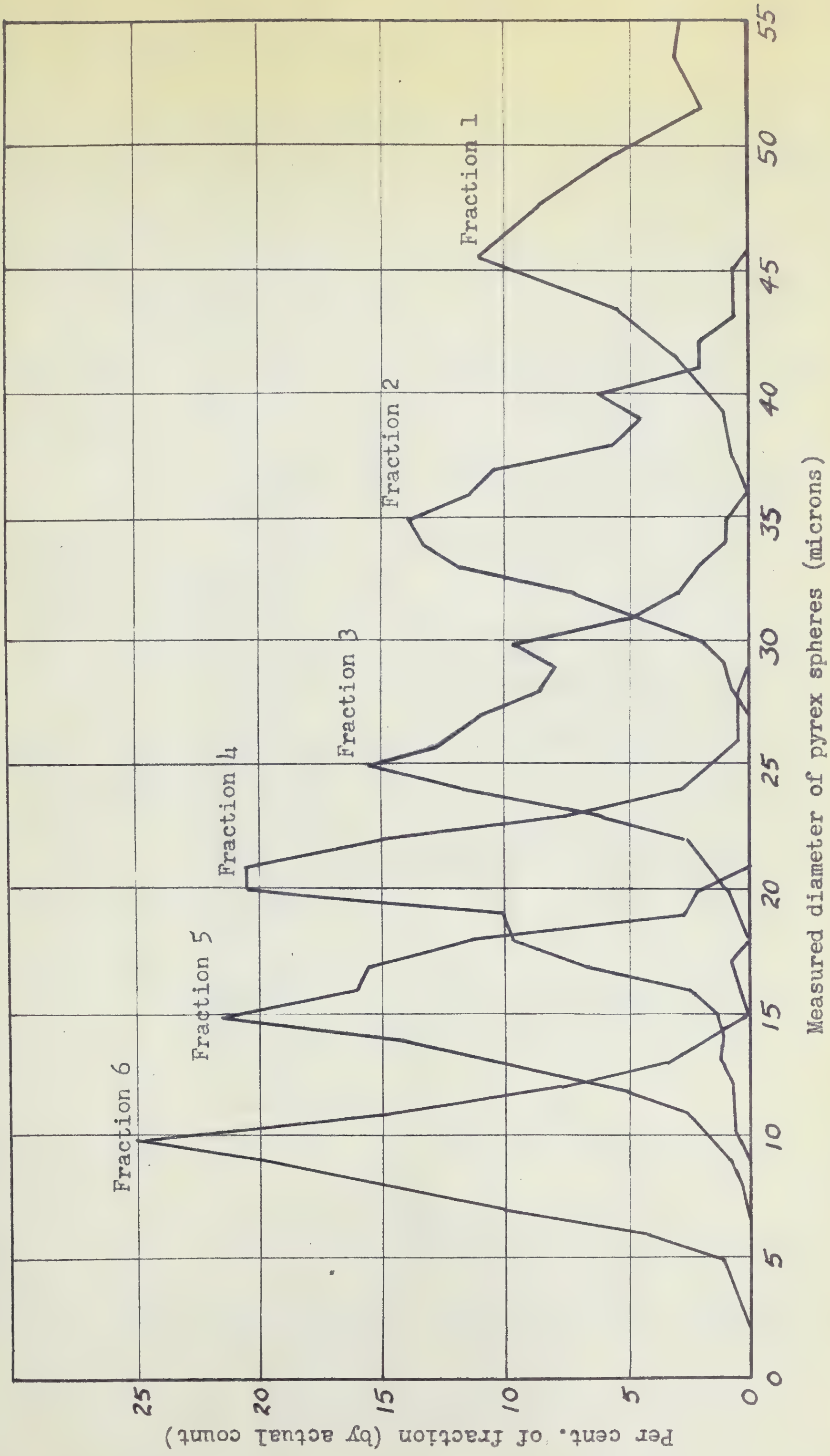


FIG. 19 - SIZE DISTRIBUTION OF SPHERICAL PYREX FRACTIONS

THE AIR PERMEABILITY APPARATUS

The air permeability apparatus used was patterned after the Rigden Apparatus shown in Figure 1. However several modifications were made to make the apparatus more flexible in range, to suit the materials available, and to simplify operating technique.

The theoretical equations were useful for determination of the required dimensions. Use of the apparatus resulted in minor changes in apparatus and technique.

The final apparatus is as shown in Figures 20 and 21. Its proportions were arrived at by the following considerations:

Design Considerations

The Kozeny equation for specific surface calculation from air permeability data was assumed sufficiently accurate for use in design of the apparatus. As adapted by Rigden for use with the Rigden type of apparatus it is:

$$s^2 = \frac{2\ g\ d_2}{a\ \ln \frac{h_1}{h_2}} \cdot \frac{AT}{k\ U\ L\ d_1{}^2} \cdot \frac{E^3}{(1 - E)^2}$$

where S = specific surface

A = area of cross section of powder bed

L = length of powder bed, (cm.)

U = viscosity of air, (poises)

E = fractional porosity of powder bed

k = Kozeny's constant = 5 (approx.)

a = cross section of U tube limbs, (cm^2)

h_1 and h_2 = heights of timing marks above the fluid equilibrium level, (cm.)

FIG. 20 - PHOTOGRAPH OF AIR-PERMEABILITY APPARATUS

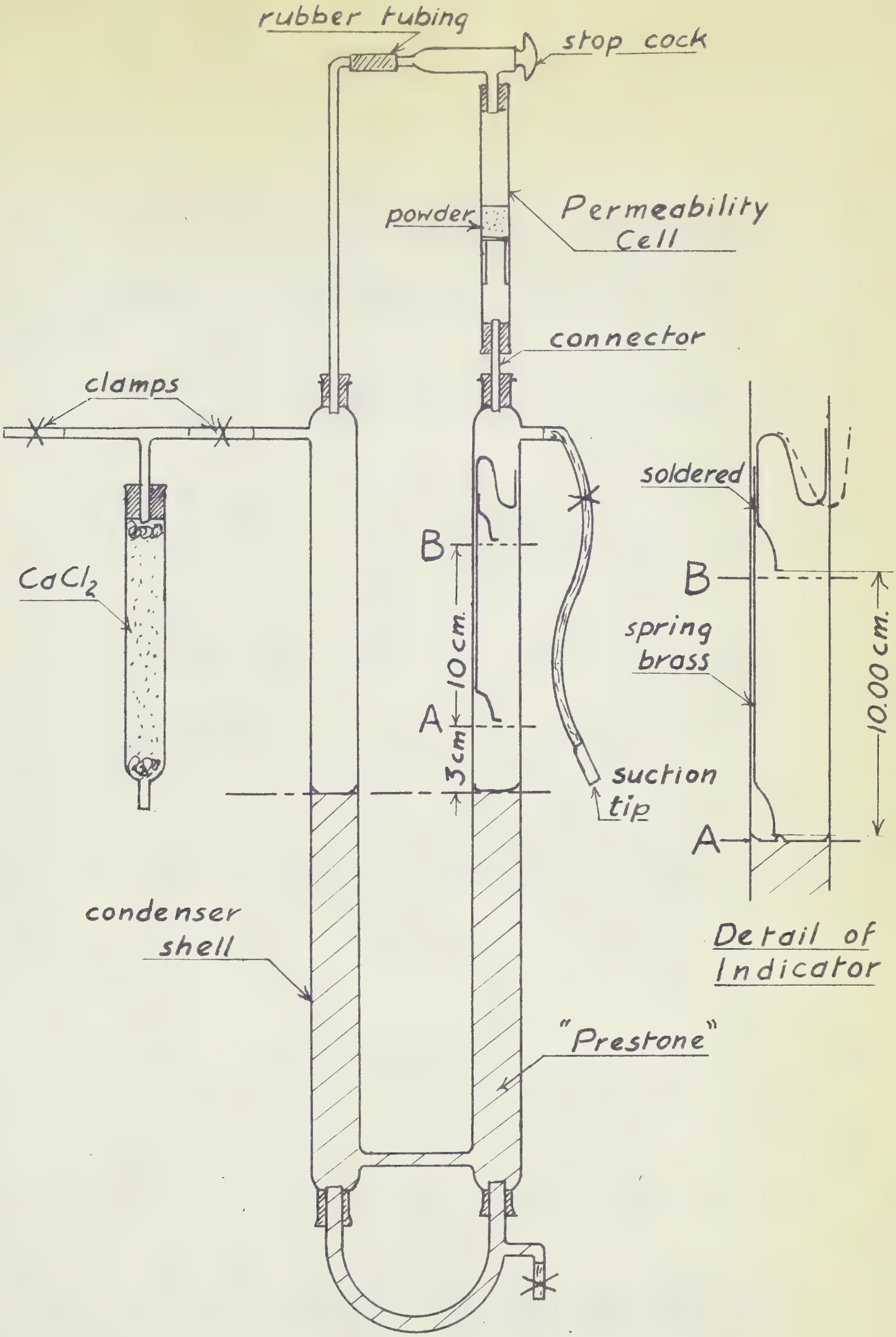


FIG. 21 - AIR - PERMEABILITY APPARATUS

d_1 = density of material (gm. per $cm.^3$)

d_2 = density of U tube fluid (gm. per $cm.^3$)

g = gravitational constant = 980

T = time for liquid to fall from h_1 to h_2, (seconds)

(Rigden's derivation of this equation from Kozeny's equation is given in Appendix 1)

The practical characteristics of a suitably accurate apparatus would seem to be:

(1) Reproducibility within 3 per cent.

(2) Range of specific surface measurements covering the -200 mesh plus 7 micron powders.

(3) A time limit of 5 minutes for a single measurement.

(4) Simple operation.

(5) Simplicity of construction and portability.

The equation shows that, for a given powder bed of given porosity, time varies inversely as the bed area and directly as the bed length. Time also varies approximately with the square of the specific surface.

Rigden states that, if the simplified variable pressure function is to be used to represent the average pressure across the bed with reasonable accuracy, h_2 must be at least 3 centimeters, and h_1 must be about 13 centimeters. However the area of the U-tube can have any practical value.

From these considerations the following dimensions were decided upon:

Area of permeability cell to be approximately 2 $cm.^2$

Area of U tube to be about 7 $cm.^2$

Depth of bed to be variable, from 1 to 5 centimeters, depending upon the specific surface of the powder.

This design gives a calculated time of 20 seconds for a 200 mesh pyrex fraction with bed depth of 5 centimeters. For a pyrex fraction averaging 7 microns in diameter, the time is 400 seconds with a bed depth of 1 centimeter.

Apparatus:

U-tube: The U-tube is formed from two condenser shells, as shown in Figures 20 and 21, which are joined at the bottom to a curved connecting tube by two rubber corks. The vertical tubes have a cross-section of 7.22 square centimeters and are 50 centimeters in length. The connecting tube is bent from 9 millimeter glass tubing with short side arm to facilitate adjustment of the fluid level. The two condenser shell side arms at the bottom are joined by rubber tubing. The top side arms act as air connections for operation. The permeability cell system is connected into the top of the vertical tubes by rubber corks.

Fluid: The fluid which half fills the U-tube is "Prestone" (ethylene glycol). This was chosen since it has a low vapour pressure, no fumes, fairly low viscosity, and no corrosive effect on the rubber connections. Water was tried but the level required constant adjustment due to evaporation. Light mineral oil gave off objectionable fumes, and also attacked the rubber corks.

To protect the fluid against absorption of water vapour from the air, the calcium chloride drying tube is connected to the intake side arm and all intake air must pass through this.

length of bed to be variable, from 1 to 8 centimeters, [illegible] upon the specific surface of the powder.

This design gives a calculated time of 18 seconds for a [illegible] mesh pyrex fraction with a bed depth of 4 centimeters. For a pyrex fraction averaging 7 microns in diameter, the time is 40 seconds with a bed depth of 1 centimeter.

Apparatus:

U-tube: The U-tube is formed from two condenser shells, as shown in figures 50 and 51, which are joined at the bottom to a curved connecting tube by two rubber corks. The vertical tubes have a cross-section of 7.25 square centimeters and are 50 centimeters in length. The connecting tube is bent from 8 millimeter glass tubing, with short side arms to facilitate adjustment of the fluid level. The two condenser shell side arms at the bottom are joined by rubber tubing. The top side arms act as air connections for operation. The permeability cell system is connected into the top of the vertical tubes by rubber corks.

Fluid: The fluid which half fills the U-tube is "Prestone" (ethylene glycol). This was chosen since it has a low vapor pressure, no fumes, fairly low viscosity, and no corrosive effect on the rubber connections. Water was [illegible] adjustment due to evaporation. Light oils [illegible] fumes, and also attacked the rubber corks.

To protect the fluids against [illegible] the air, the calcium chloride drying tube [illegible] side arm so all intake air must pass through [illegible].

Indicator: Two timing marks, A and B, are established on the right hand tube, 3 centimeters and 13 centimeters above the equilibrium level of the fluid. Due to the slow rate of fall with samples of high specific surface, it is very difficult to judge the time at which the liquid meniscus passes the lower timing mark. A timing indicator was constructed of thin spring brass, and inserted into the tube as shown in Figure 21. Two strips of brass are bent and soldered together, with the timing points exactly 10 centimeters apart. The device is then pushed to the desired position in the U-tube and is held in that position by the curved spring of the upper strip. As the liquid falls slowly past the timing point the meniscus clings to the point, but at a short fixed distance below the tip, the drop breaks. If the indicator is adjusted carefully so that the drop breaks just as the surface of the liquid passes the upper timing mark, the drop will break from the second point exactly 10 centimeters lower. Indication is thus positive and very accurate.

Timer: An electric "Time-it" timer was used, reading to 0.01 minutes. A calibration curve was used to convert time to seconds.

Permeability Cell: The accuracy of the apparatus is dependent to a large extent upon the accuracy of the dimensions of the powder bed contained by the permeability cell. The permeability cell, Figure 22 a, is patterned after the cell used by Blaine (46)(47). It is constructed of a thick walled glass tube with an internal cross-section of 2.11 square centimeters. A ledge for support of the bed is formed by an inner tube, which makes a sliding fit with the outer tube, and is cemented into place. The perforated porcelain disc which rests on the

[illegible]

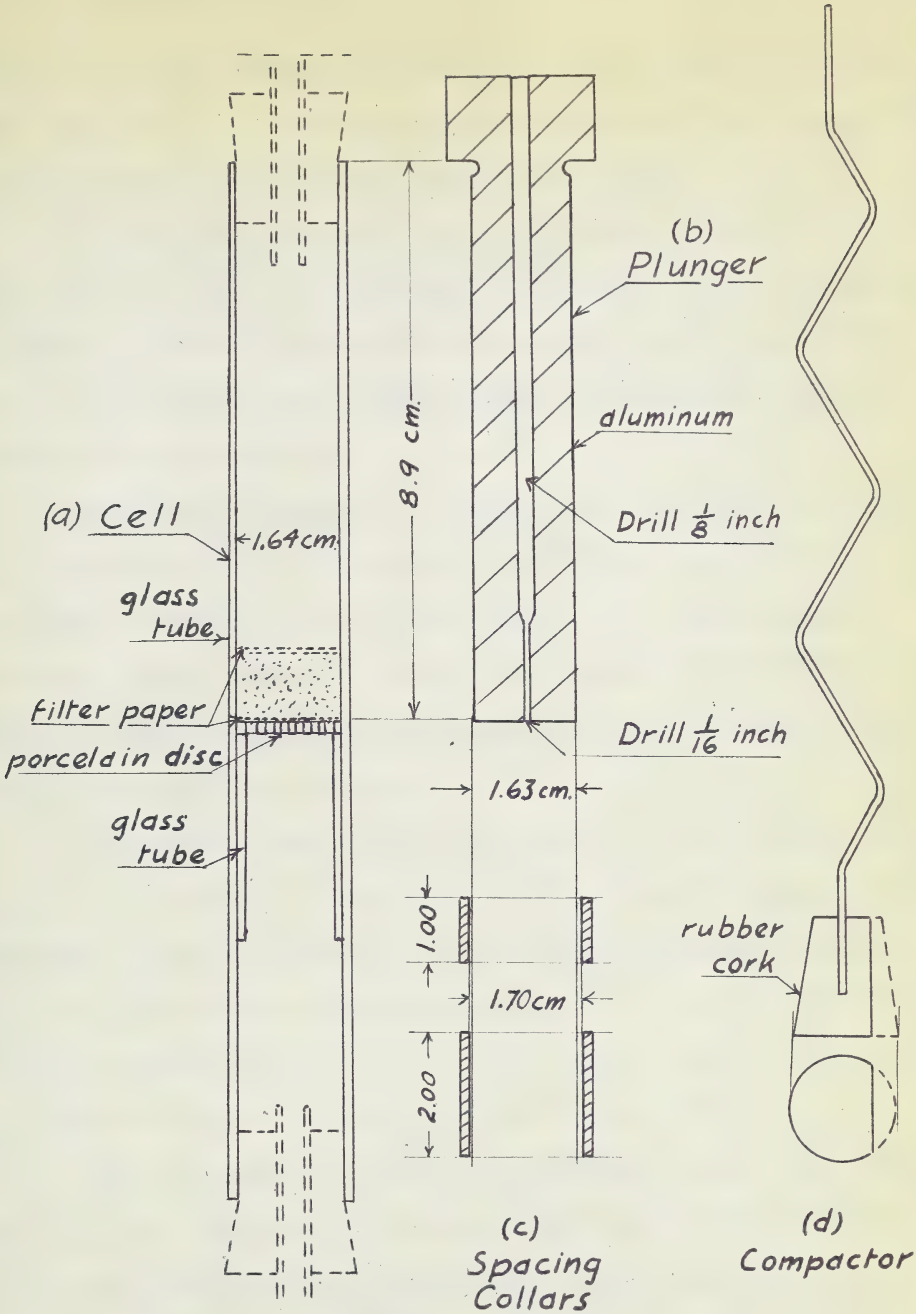


FIG. 22 - PERMEABILITY CELL AND COMPACTION APPARATUS

ledge is made from a "Coors" filter disc which is ground to fit the large tube. It is ground down to fit snuggly on the inner ledge, using glass grinding compound, and is cemented into place.

The cell is connected to the right limb of the U-tube by a short connector made of two corks and a short section of glass tubing. To the top is fitted a stop cock which is connected through a glass tube to the left hand limb of the U-tube. The cell may be quickly detached from the apparatus.

Compaction Apparatus: The powder bed is compacted into the cell with the metal plunger shown in Figure 22 b. One filter paper disc is placed below the bed and another above the bed. The plunger is accurately made so that its shoulder just touches the top of the cell when the bottom is resting on two pieces of filter paper placed upon the perforated disc in the cell.

Depth of the powder bed is controlled by brass spacing collars placed on the plunger. These are accurately ground to lengths of 1.00 centimeter, and 2.00 centimeters (Fig. 22 c). When the 1 centimeter collar is placed on the plunger the depth of the compacted bed will be 1.00 centimeters with the two filter paper discs in place. Depths of 1, 2 and 3 centimeters can therefore be produced accurately.

A second method, used only if sufficient powder is not obtainable for a standard bed depth, is to measure the distance between the top of the tube and the plunger shoulder with vernier calipers. This involves additional calculations.

In preparing the bed, a compactor designed by Gooden[44] was employed. This was used to press the powder into place and also was useful as a "policeman" for scraping down clinging powder from the

ledge is made from a "Coors" filter disc which is ground to fit the [illegible]
large tube. It is ground [illegible] to fit snugly on the [illegible]
[illegible]

The cell is connected [illegible] by a
short connector made of [illegible] and a short section of [illegible] tubing.
To the top is fitted [illegible] connected [illegible]
tube to the left [illegible]. The cell [illegible]
detached from the apparatus.

<u>Compaction Apparatus</u>: The powder bed is compacted [illegible]
cell with the metal plunger shown in Figure 22 b. One filter paper disc
is placed below the bed and another above the bed. The plunger is
accurately made so that the shoulder just touches the top of the cell
when the bottom is resting on two pieces of filter paper placed upon
the perforated disc in the cell.

Depth of the powder bed is controlled by brass spacing collars
placed on the plunger. These are accurately ground to lengths of [illegible]
centimeter, and 2.00 centimeters (Fig. 22 c). When no [illegible]
collar is placed on the plunger the depth of the compacted material is
1.00 centimeters with the two filter paper discs in place. Depths of
1, 2 and 3 centimeters can therefore be produced accurately.

A second method, used only if sufficient powder is not available
for a standard bed depth, is to measure the distance between the top
of the tube and the plunger shoulder with vernier calipers. [illegible]

[illegible]

In preparing the bed, a [illegible]
employed. This was used to pass the powder [illegible]
[illegible] a "policeman" for scraping [illegible]

sides of the cell. It was constructed as shown in Figure 22 d by cutting a section from the side of a rubber cork of correct size, and inserting a spring handle of piano wire bent to the shape shown.

Procedure:

The standard procedure for making a specific surface determination is as follows:

Bed Preparation: The cell is carefully weighed to 0.01 gram with the two filter paper discs. A rectangular piece of rubber sheet fitted around the cell prevents it from rolling on the balance pan. With the lower filter disc in place, powder is added to the cell in increments, pressing down each increment with the rubber compactor. Moderate pressure is used on the wire spring handle. Powder is added until it is just above the required depth. This depth may be conveniently indicated by a mark scribed on the side of the tube. Then the walls are scraped down and the top of the powder is levelled by holding the cell vertical and tapping it gently on the table. The levelled top should be slightly above the mark. The upper filter paper disc is then placed in the cell and pressed into place with the metal plunger. With the appropriate spacing collar on the plunger, a few taps of the cell on the table should be necessary to bring the shoulder and spacer into contact with the top of the tube. The right amount of powder is determined by trial and error, but with a bit of practice the correct amount can be judged the first time.

The exact degree of porosity obtainable with a given sample varies, within a certain range, depending on the pressure exerted on the plunger and the tapping required. When this range is known a

simpler method is to weigh out the approximate amount of powder required, place it in the cell, compact by tapping, and finish off to the exact depth with the plunger.

After the bed is prepared the cell is weighed and the weight of powder used is determined to 0.01 gram.

Permeability Test: The cell is placed on the permeability apparatus and connected up, with the stop cock above the cell closed. The clamp on the intake side arm is loosened. The clamp on the indicator side is loosened, and suction is applied to the tube until the liquid level rises about 1 centimeter above the top indicator. Then both clamps are closed tightly.

The stop cock is opened and the timer is started as the meniscus breaks from the top indicator tip. When the meniscus breaks from the lower indicator tip, the timer is stopped and the time, T, noted.

Calculations: The porosity of the bed must be calculated from its dimensions, the density of the powder, and the weight of the powder. The formula used is:

$$E = 1 - \frac{W}{d_1 A L}$$

where W = the weight of powder, (grams)

d_1 = the density of material, (grams per $cm.^3$)

A = area of the cell, ($cm.^2$)

L = depth of bed, (cm.)

E = fractional porosity.

This calculation is simplified by setting up an alignment chart (Fig. 23) which gives the porosity for a bed of powder weighing w grams, and 1 centimeter in depth. This may be used directly for beds 1

a[illegible] method is to weigh out the equivalent amount of [illegible], [illegible] to the cell, compact by [illegible], and [illegible] off the [illegible] depth with the plunger.

After the bed is prepared the cell is weighed and the weight of powder used is determined to 0.01 gram.

Permeability Test: The cell is placed on the permeability apparatus and [illegible], with the stopcock [illegible] the cell at [illegible]. The clamp on the [illegible] is [illegible]. The clamp on the indicator [illegible] is [illegible] until the liquid level rises [illegible] above the top indicator. Then both clamps are [illegible] tight.

The stopcock is opened and the timer is started as the meniscus breaks from the top indicator [illegible]. When the meniscus [illegible] the lower indicator [illegible], the timer is stopped and the time, t, noted.

Calculations: The porosity of the bed must be calculated from its dimensions, the density of the powder, and the weight of the powder. The formula used is:

$$\epsilon = 1 - \frac{W}{d_t A l}$$

where W = the weight of powder, (gram)

d_t = the density of [illegible], (grams per [illegible])

A = area of the cell, ([illegible])

l = depth of bed, ([illegible])

ϵ = fractional porosity.

This calculation is simplified by [illegible] (Fig. [illegible]) which gives the porosity for [illegible] and 1 centimeter in depth. This [illegible].

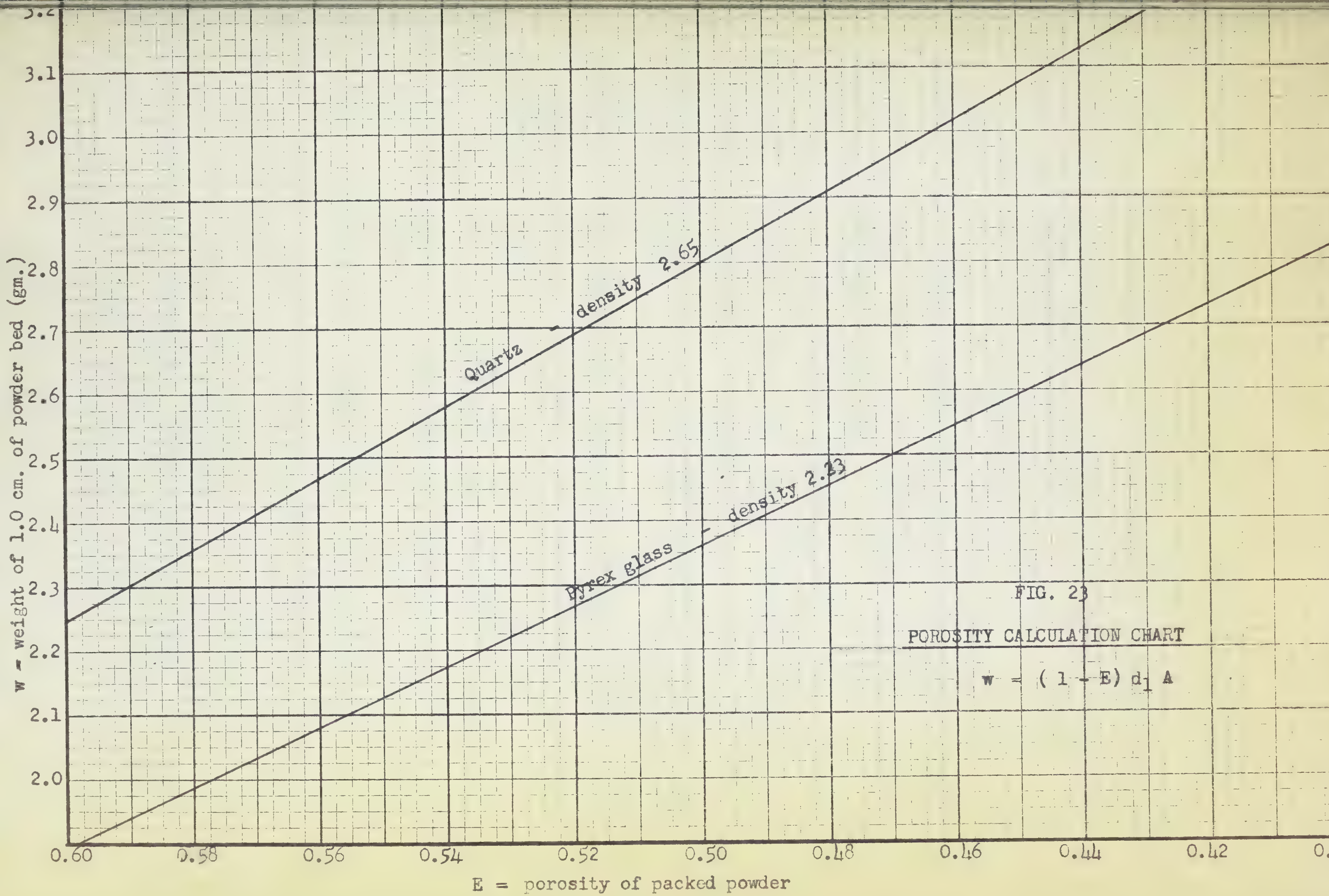


FIG. 23

POROSITY CALCULATION CHART

$w = (1 - E)\, d_1\, A$

centimeter in depth, but for beds of any other depth the weight W must be divided by L to get w, before using the chart.

Since the time T varies directly with the length of the bed L, all time values are converted to a common reference by calculating the time per unit depth of bed:

$$t = \frac{T}{L} \text{ seconds}$$

From the formula the time will vary approximately as the specific surface. Therefore the square root of t is calculated for tabulation and plotting.

Tests on Reproducibility of Apparatus

To test the reproducibility of the apparatus, a series of tests were made using Spherical Powder Fraction 3. For each test the powder was sampled, and compacted to a bed depth of 2 centimeters. The value of E was determined, and T measured on the apparatus. The time per unit bed depth, t, and $\sqrt{t}$ were calculated. Tests were conducted over a period of 6 days at temperatures ranging from 20 deg. to 25 deg. C., and are listed in Table 9 in the order in which they were made. The results are plotted in Figure 24.

In Tests 1 to 23, the samples were compacted by the standard method described above. In tests 24 to 30, compaction was by the Blaine method(46)(47), in which the weighed quantity of powder is placed in the cell and compacted with one push of the plunger without tapping. Figure 24 shows that this procedure gives more erratic values than the standard compaction method.

In Figure 24 the mean value for $\sqrt{t}$ at any porosity is indicated by the straight line. The mean deviation in $\sqrt{t}$ from this line for all samples compacted by the standard method (excluding Tests 24 to 30) is 0.031. The maximum deviation is 0.09.

Table 9 - Permeability Tests on Spherical Powder Fraction 3

Test	E	t	$\sqrt{t}$	Deviation from Plotted Mean		Mean Deviation
1	0.400	61	7.81	.04	-0.5 %	
2	0.410	56	7.48	.00		
3		rejected, non uniform porosity				
4		error in timer reading				
5	0.404	60.5	7.75	.04	+0.5 %	
6	0.408	57	7.52	.01		
7		error in timer				
8	0.406	58.5	7.65	.02		
9	0.399	62	7.87	.03		
10	0.399	62.5	7.90	.02		
11	0.385	69	8.31	.00		
12	0.390	69.5	8.33	.09	+1.1%	
13	0.399	62.5	7.90	.02		
14	0.395	64	8.00	.04	-0.5%	
15	0.403	59	7.68	.06	+0.8	
16	0.397	63.5	7.97	.02		
17	0.392	66.5	8.17	.02		
18	0.392	65.5	8.10	.05	-0.6%	
19	0.432	44.5	6.68	.01		
20	0.418	51.5	7.18	.00		
21	0.407	57.0	7.55	.05		
22	0.408	57.5	7.59	.00		
23	0.408	56.0	7.48	.09	-1.2 %	0.031
24	0.400	65.5	8.10	.25	+3.1	
25	0.398	67	8.20	.28	+3.4	
26	0.396	62.5	7.90	.10	-1.2	
27	0.407	64	8.00	.40	+5.0	
28	0.397	65.5	8.10	.14	+1.7	
29	0.408	58.5	7.65	.12	+1.6	
30	0.401	61	7.81	.00	+0.0	

[illegible]

[illegible]

[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]				
	[illegible]				
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		[illegible]			[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
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[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

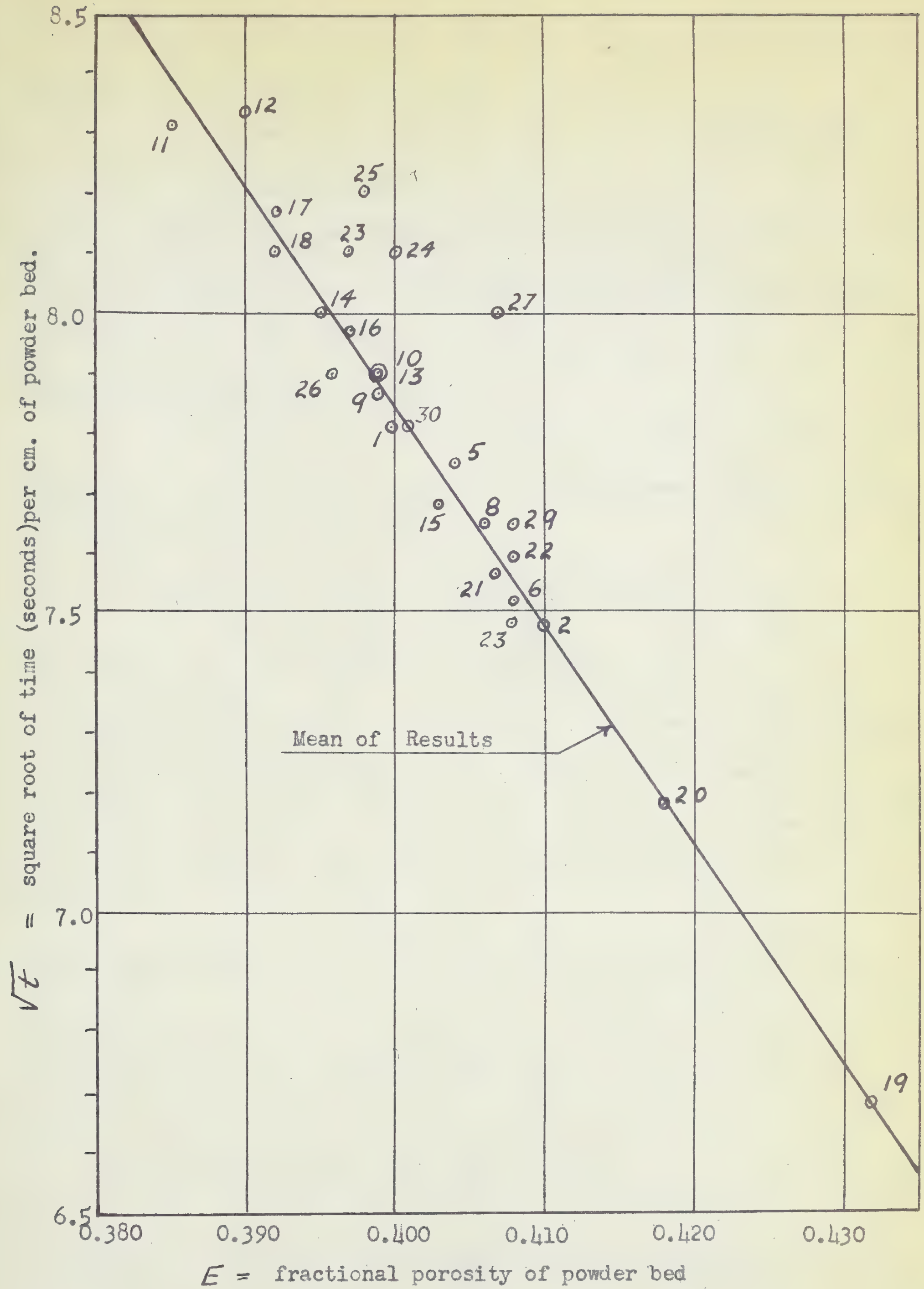


FIG. 24 - PERMEABILITY TESTS ON SPHERICAL FRACTION NO. 3

The standard deviation is 0.041 and the maximum allowable deviation computed by the method given by Taggart[61] is 0.09.

The probable error computed by the approximate formula[60] is:

$$0.031 \times 0.8453 = 0.027$$

Computed on a percentage basis, the probable reproducibility is from 0.4 to 0.33 per cent. over the range of porosities tested, while the maximum error varies from 1.3 to 1.0 per cent.

Calculation of Experimental Error

In order to determine the possible error in the results of permeability tests with the apparatus on a given powder, a calculation of experimental errors was made.

From Rigden's equation it may be seen that

$$t \propto \frac{\ln h_1/h_2}{d_2} \times U \left\{ \frac{E^3}{(1 - E)^2} \right\}$$

other factors being constant for a series of tests on a given material.

The variable factors are the result of inaccuracy in determining the porosity of the bed, systematic errors in time measurement due to change of temperature, and errors in timing.

The error in porosity is the result of an error in powder weight and an error in bed depth measurement, combined with the error in reading the porosity calculation chart. Assuming a weight of powder of approximately 2.50 grams for a depth of 1.0 centimeters, at a porosity of approximately 0.47,

Error in powder weight 0.001 gm. = .05%

Error in bed depth 0.005 cm. = .50%

Error in weight per cm. of packing= .55% or 0.013 gm.

The resulting error in porosity from the chart assuming negligible

error in plotting is ± .0025 or ± 0.5%. The resulting error in the porosity function is therefore ± 0.5%.

Since the temperature was 22.5° C. ± 2.5°, this will cause an error in the volume and density of the manometer fluid, and the viscosity of the air. At 20°C. the coefficient of expansion of ethylene glycol is 0.5×10^{-3}. The fluid volume in the apparatus is 340 ml. Therefore the change in volume for a temperature change of 2.5°C. will be 0.44 ml. The equilibrium level in the U-tube will vary 0.3 cm. causing a variation of 0.03 cm. in h_1 and h_2. The error in $\ln h_1/h_2$ will then be ± 0.0023 cm. or ± 0.15%. For the same variation of temperature the density of the fluid will vary ± 0.13%. The combined effect of variation in temperature on the mean pressure across the powder bed will then be ± 0.28%.

With an increase in temperature of 5 degrees C. in this range the viscosity of air increases 3.0%. Therefore the viscosity of air, U, will vary ± 1.5% for a temperature variation of ± 2.5 degrees C.

The error in timing the fall of the liquid is ± 0.5 seconds or ± 1.0% for a 50 second run.

Combining the results of errors in porosity mean pressure, viscosity of air, and time, the possible error in the value of "t" is ± 0.5, ± 0.28 ± 1.5 ± 1.0 = ± 3.3%.

This is greater than the maximum error found in the tests on Spherical Fraction 3, Table 9, which was compacted by the standard method. This indicates some degree of compensation of error.

CALIBRATION CURVES FOR THE APPARATUS

The six standard powder fractions were used to set up calibration curves for the apparatus covering a range of specific surfaces from 540 to 3000 for pyrex. However since the ultimate object was to use the apparatus on mineral powders, the variable factor of density of material was eliminated from the calibration by setting up the curves for volume-surface, S_V, in square centimeters per cubic centimeter of material.

It was found that the porosity range obtainable with the pyrex spheres did not include the porosities obtainable with broken materials, so the range was extended using quartz powders.

Curves for Glass Spheres

Procedure: Permeability tests were run on Spherical Fraction 3, as has been described above. By similar procedure permeability tests were run on each of the other five standard spherical powder fractions. With each powder the widest obtainable range of uniform porosities was tested. It was found that this range was very narrow for the coarse fractions regardless of the technique of compaction; and was quite wide for fine fractions.

The values of $\sqrt{t}$ obtained at each porosity are tabulated in Table 10, and are plotted in Figure 25. From this plotted data were derived the calibration values required for construction of the calibration curves.

A best fitting line is drawn for each fraction, which indicates the mean relationship between $\sqrt{t}$ and E at the given surface value, and for the range of porosities shown.

Table 10 - Permeability Tests on Spherical Powders

Fraction	Porosity E	$\sqrt{t}$
1	.391	4.95
	.401	4.53
	.409	4.36
2	.407	6.08
	.408	5.97
	.414	5.70
	.391	6.45
	.388	6.67
	.398	6.21
	.410	5.78
4	.460	7.67
	.444	8.31
	.420	9.06
	.438	8.64
	.432	8.66
	.422	9.1
5	.418	11.6
	.425	11.2
	.436	10.9
	.437	10.8
	.444	10.3
	.440	10.8
	.412	12.0
	.447	10.4
	.434	10.6
	.428	11.1
	.425	11.4
	.418	11.6
	.412	11.7
6	.492	12.23
	.494	12.05
	.478	12.67
	.474	13.00
	.461	13.55
	.455	14.1
	.448	14.6
	.437	15.4
	.436	15.5
	.431	15.75
	.431	15.75
	.423	16.1
	.411	17.2
	.404	17.35
	.398	18.00
	.405	17.4
	.405	17.7
	.410	17.3
	.550	8.55
	.535	9.74
	.480	12.65
	.505	10.8

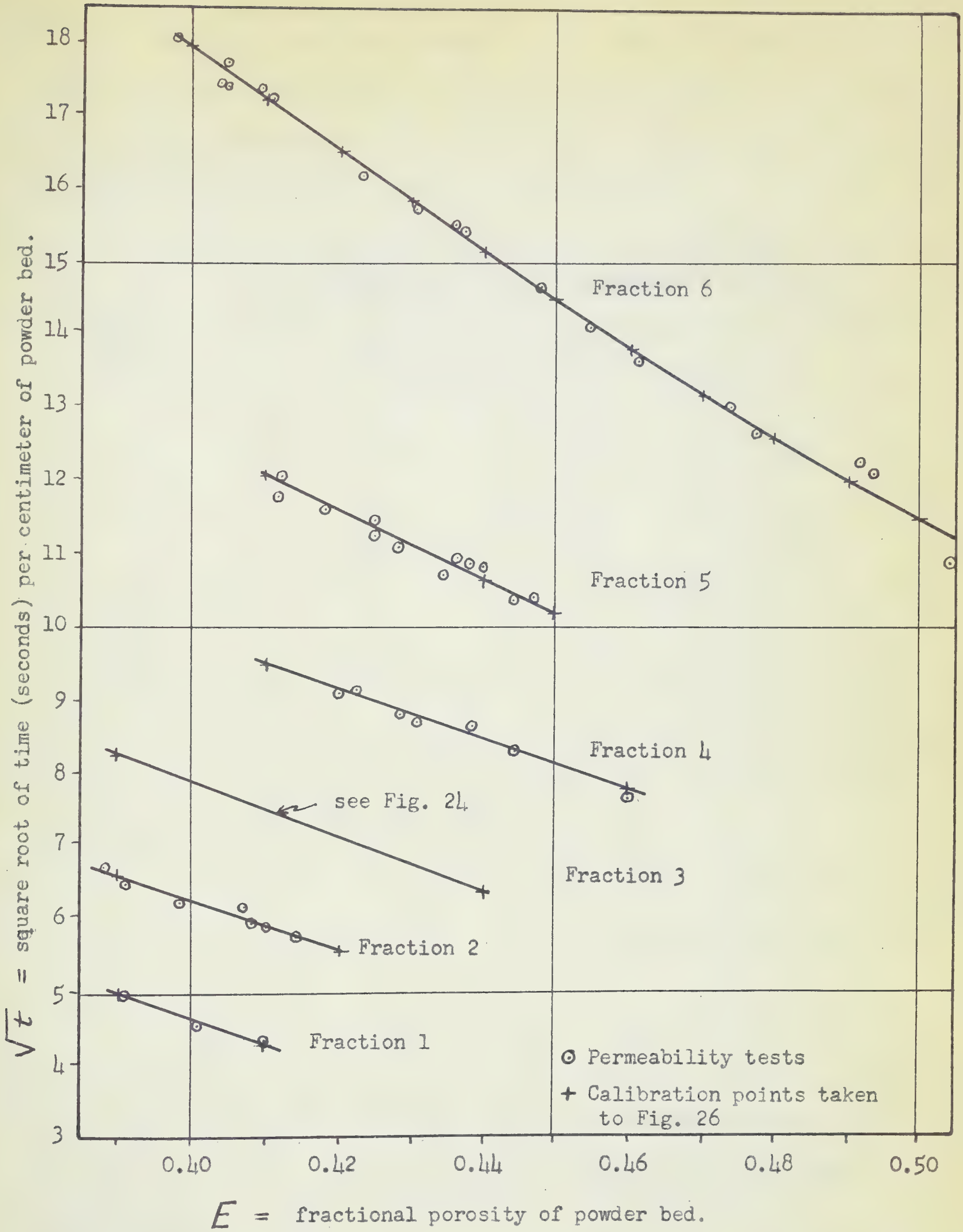


FIG. 25 - PERMEABILITY DATA FOR CALIBRATION OF APPARATUS USING SPHERICAL PYREX POWDERS.

With the exception of the line for Fraction 6, which must be slightly curved, the best fitting mean lines are straight. The probable error in the plotted mean values, computed as for Fraction 3, is given in Table 11.

Table 11 - Probable Error in Plotted Mean Values for Permeability Results on Spherical Fractions

Spherical Fraction	Mean Deviation in $\sqrt{t}$	Probable error in $\sqrt{t}$	Probable error %
1	.030	.026	0.6
2	.060	.055	1.00
3	.031	.027	0.34
4	.076	.064	0.70
5	.12	.10	0.9
6	.158	.134	0.9

Calibration values of $\sqrt{t}$ and E are taken from the mean line for each fraction and are plotted on the Calibration Chart, Figure 26, against the appropriate value of S_v. This gives a set of porosity-time points for each fraction. Then points of equal porosity are joined by smooth curves. The resulting calibration curves give the time for any given porosity and surface value.

Also, if the porosity and time for any sample of powder has been determined by the apparatus, the surface of the powder, S_v, can be taken from the calibration curves. When S_v is thus determined, the specific surface, S, of the powder, can be calculated by dividing S_v by the material density.

Since the porosity curves are plotted at 0.01 intervals, interpolation is necessary for porosities determined to thousandths.

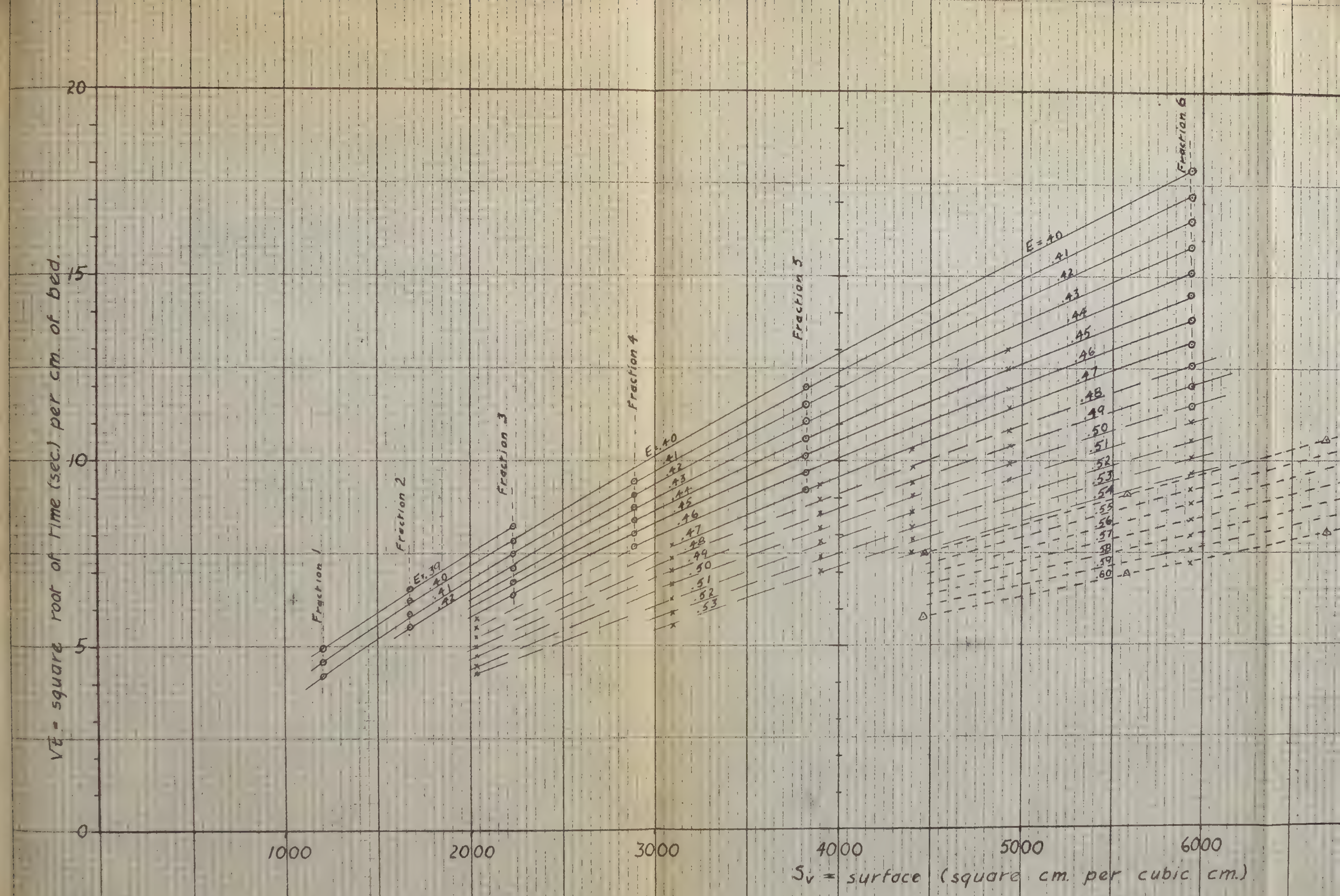


FIG. 26 - CALIBRATION CHART FOR APPARATUS

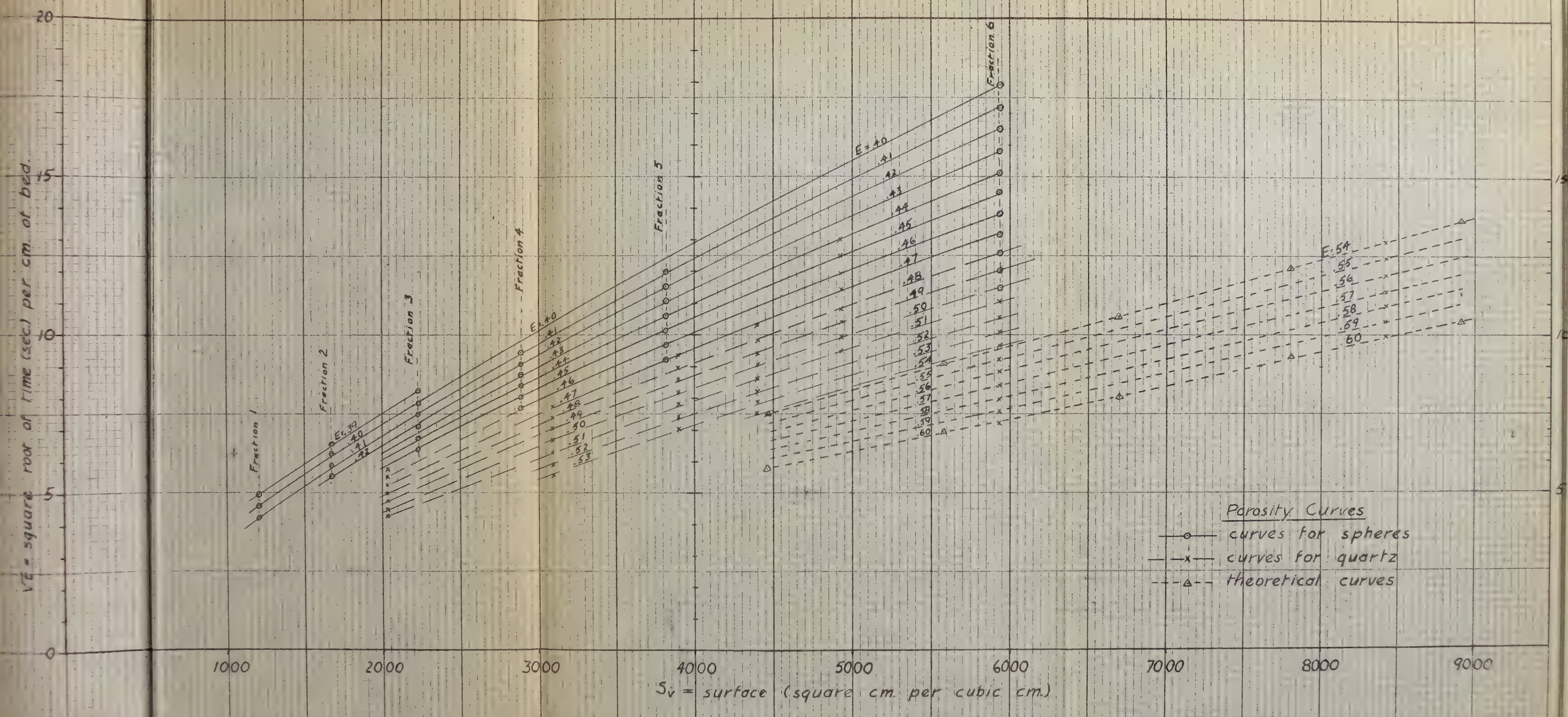


FIG. 26 - CALIBRATION CHART FOR APPARATUS

Tests of Accuracy: As a check on the accuracy of the calibration curves and reproducibility of the apparatus, a composite sample was made up of Fraction 4 and Fraction 5, 50 per cent. by weight of each being thoroughly mixed. Two runs were made with the following results:

Table 12 - Results of Surface Determinations on a Composite Spherical Powder.

Test	Porosity	t	$\sqrt{t}$	S_v	S
1	.411	110 sec.	10.5	3260	1460
2	.415	108	10.4	3290	1475
			Average		1468
			Calculated Value		1500
			Deviation		2.1%

Theoretically, extrapolation of all lines in Figure 26 to zero time, should give zero surface at any porosity. If the curves are produced they do not pass through the origin, but intercept the $\sqrt{t}$ ordinate. This intercept is approximately the filter paper correction, for the two pieces of filter paper. A test was carried out to determine the actual filter paper error which displaces the curves.

Four time measurements were made on a standard 2 centimeter bed (S_v = 5000) with results differing by less than 0.5 seconds and averaging 46 seconds. An additional filter paper was then pressed in place by the plunger, at the same time placing a filter paper spacing ring under the plunger shoulder so that the depth of the powder bed was not changed. Four time measurements were made then with results averaging 50 seconds. The correction in T for one filter paper equals

4 seconds, or a correction of 2 seconds in t, the time for unit length of packing. Therefore the two filter papers used in the standard bed should give a paper correction of 4 seconds in any value of t. Subtraction of 4 seconds from the plotted values for t for Fractions 1 and 2, gives curves which pass through the origin. The calibration curves must therefore be nearly correct.

Extension of Porosity Range with Quartz Powders

Preliminary attempts to use the sphere calibration curves for surface determination of angular material, were not satisfactory since the spherical curves do not cover the normal porosity range for packed powders of sized angular particles. Only in the case of the coarse fractions or unsized powders could the material be packed to a porosity low enough to fall on the spherical curves. Therefore extension of the porosity range covered by the curves was necessary. This was carried out and checked using quartz powders.

Preparation of Quartz Powders: The quartz powders for use in calibrating the air permeability apparatus were prepared from Ottawa Sand, by grinding in a ball mill, cleaning with acid, and sizing in the elutriator. The grinding was done, dry, in a 12 inch laboratory ball mill using 10 kilograms of cast iron balls. The sand charge was 400 grams. Time of grinding was 20 minutes.

The mill product was screened with the following results:

+ 100 mesh	0.5%
+ 150	5.0
+ 200	30.4
-200	64.1

The -200 mesh product was treated by stirring for one hour with 50 per cent. hydrochloric acid to dissolve abraded mill iron. The mixture was then diluted with water and after 3 hours the dilute acid

was siphoned off. The quartz powder was suction filtered in a 4 inch Buechner funnel, and washed repeatedly with tap water until tests of the filtrate with methyl orange showed no trace of acid. The clean quartz powder was then dried.

Using a similar procedure, a 400 gram sample of Ottawa sand was ground 10 minutes, screened, and treated with acid to obtain a -200 mesh product. The screen analysis was:

+65 mesh	7.2%
+100	16.0
+150	21.0
+200	17.5
-200	38.3

Elutration of two 50 gram samples, designated Q II and Q III, from the cleaned 20 minute grind product, was carried out using the same apparatus and standard procedure as described above for glass spheres. The quartz fractions were filtered in gooch crucibles, weighed, and dried.

Two 20 gram samples of the 20 minute grind product, Q V and QVI, were elutriated to obtain two +7.2 micron, -200 mesh, quartz powders. Tubes 1 and 4 were used with a flow rate of 16 millilitres per minute, and with elutriation procedure as described for Stage 3 for sphere elutriation.

Two 20 gram samples, QIX and QX, from the 10 minute grind -200 mesh product, were also scalped by elutriation as for QV and QVI.

The resulting quartz powders are tabulated in Table 13.

Curves for Quartz Powders: Permeability tests were run on the quartz powders varying the range of porosity from the minimum obtainable to the highest possible uniform porosity. The standard method for compaction, as described for spherical powders, was used.

Table 13 - Results of Preparation of Quartz Powders for Calibration

Sample	Grind	Elutriation Fraction	Powder Fraction
Q I	20 min.	1	Q 9
		2	Q 10
		3	Q 11
		4	Q 12
		5	Q 17
		6	Q 18
		7	O'flo
Q II	20 min.	1	Q 13
		2	Q 14
		3	Q 15
		4	Q 16
		5	Q 20
		6	Q 21
		7	O'flo
Q V	20 min.	+7.2 micron	
Q VI	"	+7.2 "	
Q IX	10 min.	+7.2 " (nominal)	
X	"	+ 7.2 "	

The density of the material was determined by specific gravity bottle on the coarsest fraction. The density, 2.65 grams per cubic centimeter, was assumed for all fractions. Porosities were determined using the alignment chart, Figure 23.

The range of porosities for the quartz powders and the measurements of $\sqrt{t}$ made at each porosity are tabulated in Tables 14 and 15.

To obtain values for the calibration curves, the results from Table 14 were plotted as shown in Figure 27. A best fitting line was drawn for each powder. The value of S_V was determined for

Table 14 Permeability Tests on Quartz Powders Used for Calibration Curves

Powder	E	$\sqrt{t}$	S_v from Sphere Curves
Q 13	.50	4.36	
	.515	4.00	
	.485	4.85	
	.465	5.35	
	.445	5.80	2030
Q 10	.515	5.65	
	.525	5.47	
	.53	5.29	
	.515	6.33	
	.475	7.50	3100
	.485	7.28	
	.492	6.9	
Q IX	.475	9.16	3900
	.465	9.70	
	.48	8.60	
	.49	8.5	
	.50	7.92	
	.507	8.0	
	.52	7.55	
	.525	7.2	
	.53	7.0	
Q 15	.54	7.42	
	.54	7.42	
	.47	10.1	4400
	.505	8.72	
	.505	8.49	
	.50	9.37	
	.49	9.47	
	.51	8.6	
	.525	8.12	
	.54	7.56	

Powder	E	$\sqrt{t}$	S_v
Q 11	.48	9.9	4400
	.49	9.32	
	.515	8.32	
	.520	8.12	
	.54	7.5	
	.55	7.15	
	.535	7.55	
Q V	.505	9.75	
	.490	10.4	
	.455	12.4	
	.445	12.8	4920
	.46	12	
	.485	10.4	
Q 12	.58	7.62	
	.555	9.06	
	.54	9.80	
	.525	10.3	5940
Q 16	.555	9.17	
	.525	10.1	
	.505	11.3	5940
Q 20	.60	9.9	8400
	.60	9.85	
	.59	10.6	
	.565	11.7	
	.53	13.4	
	.515	14.15	

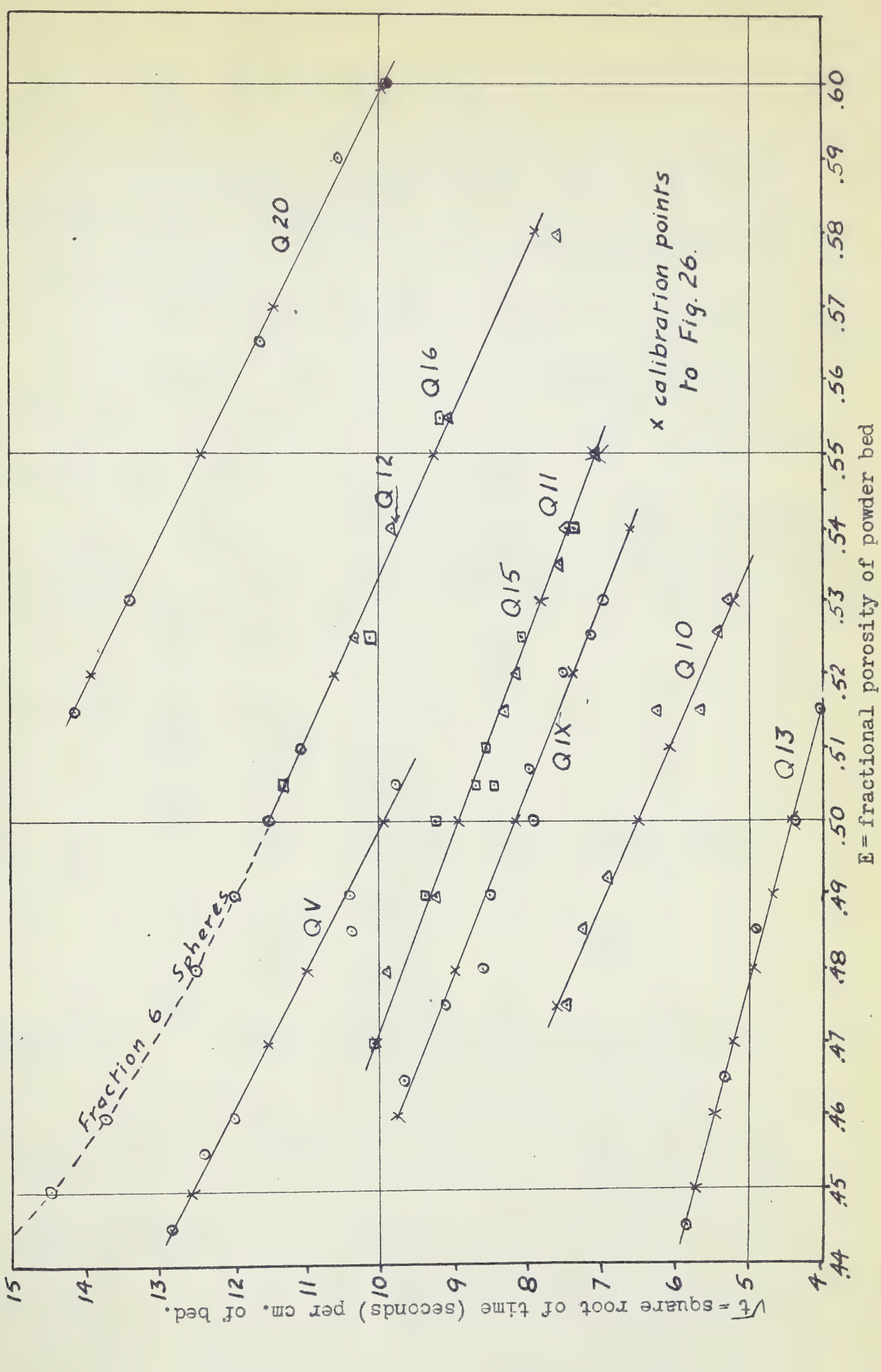


FIG. 27 - PERMEABILITY TESTS ON QUARTZ FRACTIONS

each powder from Figure 26, if the porosity range extended on to the sphere curves. The additional porosity curves were plotted as shown using the time-porosity points from the plotted mean lines in Figure 27.

The resulting calibration curves covered the range of porosities normally obtainable with the ground mineral products used in later experiments.

Table 15 shows the reproducibility of the calibrated apparatus in determining the surface of six quartz fractions. The maximum deviation from the mean of any one sample is 4.7 per cent. with the greatest mean deviation being 2.7 per cent.

Table 15 - Surface Determinations on Quartz Fractions

Fraction	E	$\sqrt{t}$	S_v from chart	Deviation from mean	Deviation %	Probable % error	Standard Deviation
Q VI	.475	9.8	4300	180	4.0		
	.455	11.0	4400	80			
	.49	9.37	4400	80			
	.51	8.65	4500	20			
	.525	8.00	4500	20			
	.545	7.30	4500	20			
	.50	9.25	4550	170			
	.455	11.8	4670	210	4.7		
		mean	4480	98	2.2	1.86	2.7
Q X	.50	8.0	3900	10			
	.515	7.48	3900	10			
	.54	6.8	4000	110	2.8		
	.43	10.95	3800	40			
	.445	10.3	3830	60			
	.465	9.5	3900	10			
		mean	3890	48	1.2	1.02	1.6
Q 9	mean of	3 tests	2070	40	2.0		
Q 14	mean of	4 tests	3120	85	2.7	2.3	
Q 17	mean of	4 tests	7910	52	0.65		
Q 18	one test .555	19.0	14500	Calculated			

Extrapolation of Curves for High Surface Values

It was discovered that the range of surface values covered was not high enough to include samples of ground materials whose specific surface was desired.

Therefore extrapolation of the curves was undertaken to include, at least approximately, these high values. Since the rate of curvature of the curves at high porosities was unknown, the correlation of theory with the calibration curves in this region was attempted.

The theoretical values were calculated from Rigden's equation, as corrected for slip by Lea and Nurse(52). Lea and Nurse show that for slip flow the rate of flow in the Kozeny type of equation must be multiplied by a correction factor:

$$C = 1 + \frac{S d_1 \, (1 - E) \, Z \, \lambda}{E}$$

as explained in the section covering theory.

The Rigden equation then becomes

$$S^2 = K \, . \, f(E) \, . \, t \, . \, \frac{1}{{d_1}^2} \, . \, C \quad ,$$

if the factors constant to the apparatus are grouped into the constant

$$K = \frac{2 \, g \, d_2}{a \, \ln \frac{h_1}{h_2}} \, . \, \frac{A}{k \, U} \quad ,$$

and f(E) is the porosity function = $\frac{E^3}{(1 - E)^2}$.

Theoretical time values for porosities of 0.40, 0.44, 0.50, 0.54, and 0.60, were calculated using this formula transposed into the form

$$\sqrt{t} = \frac{S \, d_1}{\sqrt{k} \, \sqrt{f(E)} \sqrt{C}} \quad .$$

A sample calculation is shown as Appendix II. The calculated time values for specific surfaces from 500 to 4000 for pyrex glass, are shown in Table 16, The calculated data with the corresponding calibration curves are plotted in Figure 28. In plotting the theoretical data, the values for $\sqrt{t}$ were recalculated to include the filter paper correction by the formula

$$\sqrt{t} = \sqrt{(\sqrt{t}_{theoretical})^2 + 4}$$

to bring theoretical and experimental values to a comparative basis.

It may be seen from Figure 28 that the calculated values agree very closely with experimental values for porosities 0.54 and 0.60. The theoretical curve for E = 0.60 was therefore plotted on the calibration chart, Figure 26, as a basis for extrapolation to S_v of 8920. The experimental values for quartz fraction Q 20, Figure 27, were plotted on this base to obtain other points on lower porosity curves.

From the permeability test at E = .60 and the theoretical curve for E = .60, S_v for Q 20 is 8400. Points for porosities from 0.60 to 0.52 were plotted as shown and the calibration curves previously obtained with quartz fractions were produced to pass through these new points.

An indication that the assumption of the E = 0.60 curve is justified is obtained when calculated points for E = 0.54 are plotted on Figure 26. These fall almost exactly on the calibration curve for 0.54 as extrapolated. Also as shown in Table 15, four surface determinations on Q 17, using these curves, had a mean deviation of only 0.65 per cent.

From the above it would appear that the theoretical calculation is reasonably accurate for even higher surface values at porosities around 0.60. A permeability test was therefore run on Q 18 and the surface calculated by the modified equation as 14,500 sq. cm. per cubic centimeter or a specific surface of 5500. This calculation is shown in Appendix III.

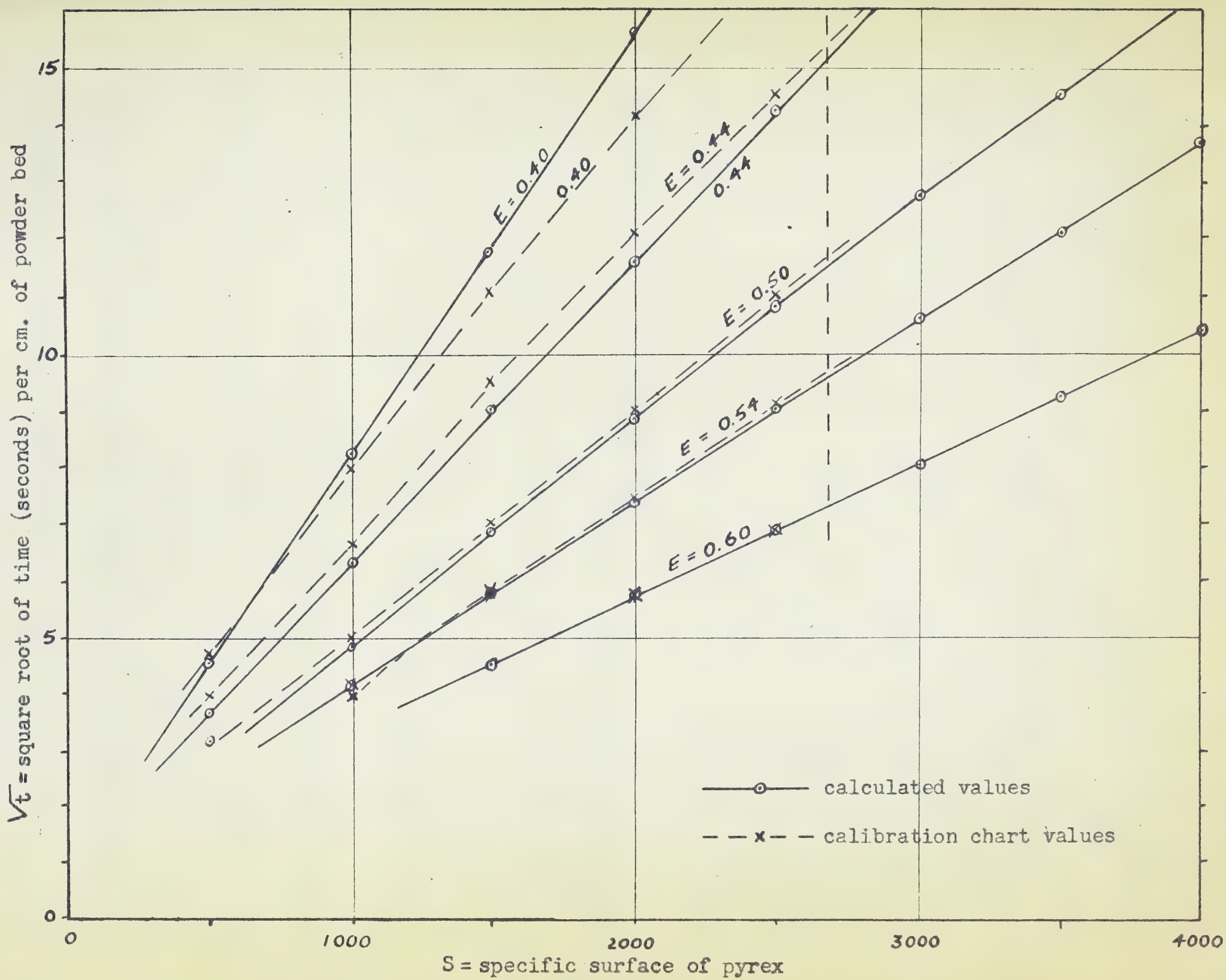


FIG. 28- COMPARISON OF CALCULATED AND CALIBRATION CHART VALUES

Table 16. Time-Porosity Values Calculated from Theory

Values of $\sqrt{t}$

Porosity S	.40	.44	.48	.50	.54	.60
500	4.04	3.05	2.54	2.26		
1000	7.92	5.96	4.89	4.42	3.68	
1500	11.60	8.80	7.25	6.57	5.42	4.06
2000	15.15	11.42	9.45	8.67	7.18	5.36
2500	18.42	14.10	11.65	10.62	8.84	6.65
3000	21.70	16.70	13.80	12.60	10.42	7.82
3600				14.35	11.90	9.06
4000				16.20	13.50	10.20

Tests on Composite Quartz Samples

Tests were run on two composite samples, made up of quartz fractions, to determine the accuracy of the air permeability method on heterodisperse samples.

For the first composite, Compo II, 2.000 grams each of Q 13 and Q 15 were mixed thoroughly. Permeability tests were run and the specific surface determined.

For the second composite, Compo III, 2.000 grams of Q 20 was added to Compo II, so that the final result contained one-third of each fraction.

The results are given in Table 17, which shows the maximum difference between the calculated and measured specific surfaces of the composites to be 2.4 per cent.

Table 17 - Tests on Composite Quartz Samples

Composite	Components			Specific Surface		
	Fraction	%	S	Calculated	Measured	Difference
II	Q 13	50	715	1197	1210	1.1%
	Q 15	50	1660			
III	Q 13	33.3	715			
	Q 15	33.3	1660			
	Q 20	33.3	3190	1854	1810	2.4%

ACCURACY OF THE CALIBRATED APPARATUS

The error involved in surface determinations made by the calibrated apparatus is the combined error inherent in the calibration curves and permeability tests.

Accuracy of the Calibration Curves

Curves for Spheres: The accuracy of the calibration curves for spheres is dependent upon the accuracy of the standard, the accuracy of the plotted curves, and the errors in interpolation.

As shown previously the surface standards can be taken as accurate within 2 per cent. The probable error in the calibration permeability values has been shown to be 1.0 per cent. (see Table 12). The calibration points for spheres as plotted on the Calibration Chart, Figure 26, may therefore be taken as having a probable error of 3 per cent. Interpolation errors in drawing the curves will not exceed 0.15, or 1.0 per cent. in the value of $\sqrt{t}$, even for specific surfaces of 5000. Therefore the curves for spheres may be taken as accurate within 4 per cent.

Curves for Angular Materials: Since the probable error in the mean calibration values taken from the results of the permeability tests on any quartz fraction, as in Figure 27, is 1.0 per cent., and since the quartz curves are based upon the sphere curves, the probable error in the quartz curves is 5 per cent.

The error in the theoretically extrapolated curves for high specific surfaces is indeterminate, since the validity of the **equation** is not proven, but will not exceed the error in experimental curves by more than 1 per cent., since the theoretical curves agree within 1 per cent. with the experimental curves within the porosity ranges and S_v values tested.

Error in Permeability Tests

For Spherical Powders: The error in reproducibility of individual permeability tests must be added to the Calibration Curve error to arrive at the final error in a specific surface determination. The reproducibility of tests on spherical powders has been shown to be 1 per cent. (Table 12). Added to the 4 per cent. error in the curves this gives a final probable error of 5 per cent.

For Angular Powders: The probable error in permeability tests on Quartz Fraction Q 14 is 2.3 per cent., as shown in Table 15. This error is reduced to 1.86 per cent. when 8 tests are made, as for Q VI. An error of 2.3 per cent. in reproducibility, when added to the error in the curves, gives a probable error of 7.3 per cent. in the specific surface determinations made on angular powders.

The higher error, in the case of angular particles, is due to the dependence of the curves for angular materials upon the sphere

curves, plus the additional error due to the greater difficulty of obtaining reproducible compaction of the angular particles.

The error in reproducibility may be reduced by repeated tests on the same sample, as for Q VI.

SUMMARY

The air-permeability apparatus as calibrated by the above procedure has a probable error of 7.3 per cent. when used for determination of the specific surface of natural powdered minerals. When tests are made on powders which are too fine to give data which falls upon the experimentally determined curves, the theoretical calculation by the modified Rigden equation should be reasonably accurate.

The accuracy of the method when used on heterodisperse powders is approximately the same as when used on sized fractions, since no greater error was found on unsized products, such as Q VI, than on sized fractions such as Q 14 (Table 15). The measured surfaces of the quartz composite samples checked with the calculated surfaces within 2.4 per cent. (Table 17).

The good agreement of the theoretical curves with the calibration curves in Figure 28 would seem to indicate that, for the porosity range 0.50 to 0.60, the tedious calibration procedure may be omitted with little loss in accuracy. As shown in Tables 14 and 15 uniform compaction can be readily obtained within this range for all quartz samples. Therefore curves can be set up from theory which should be satisfactory for all angular materials. Calibration against standard powders will still be necessary for lower porosities.

SIZE-SURFACE MEASUREMENTS ON PURE MINERALS

The second phase of the experimental investigations involved the correlation of measurement by the calibrated air-permeability apparatus with a standardized elutriation procedure, so that the distribution of surface within a -200 mesh product could be evaluated.

Accordingly investigations were carried out to determine the reproducibility of the elutriation-surface measurements, and to obtain standard calibration data for a number of pure minerals.

SIZE SURFACE MEASUREMENTS ON QUARTZ POWDERS

The data obtained on the quartz powders used in the permeability apparatus calibration, was combined with the results of additional tests on quartz powders in order to determine the reproducibility of size-surface measurements.

Procedure

Elutriation of Quartz Fractions: The quartz fractions from QI, QII, QVI, QIX, and QX, were prepared as previously described (Table 13).

Additional quartz fractions were prepared from both grinds of the Ottawa sand by elutriation.

The elutriation procedure was modified to eliminate Stage I of the standard sphere procedure. Arrival of Tube 5 (Table 1) made this possible. The modified procedure is shown in Table 18.

Stage I: Dispersal of the initial 50 gram sample is as for the first stage of sphere elutriation. In order to allow for the length of Tube 5, the time of run is extended to 2 1/2 hours. The sides of Tubes 4 and 5 must be tapped to dislodge fines. In all other respects the procedure is as for the second stage of sphere elutriation.

Stage II: Elutriation of the overflow product from Stage I is carried out exactly as for the third stage of sphere elutriation, at a flow rate of 16 millilitres per minute, and using Tubes 1 and 4.

All fractions are filtered, dried, and weighed.

Table 18 - Modified Procedure for Quartz Elutriation

Stage	Fraction	Flow Rate (ml. per min.)	Tube	Nominal Size of Glass Spheres (microns)
I	1	175	1	+ 44.1
	2		2	+ 33.7
	3		4	+ 23.7
	4		5	+ 19.2
II	5	16	1	+ 13.2
	6		4	+ 7.2
	7		o'flo	- 7.2

Surface Determinations: Surface determinations were made on each quartz fraction by the standard procedure using the calibrated air-permeability apparatus. Two tests at different porosities were run on each fraction. If the surface results agreed within 5 per cent., the mean was taken as the most probable value. Occasionally an erratic result was obtained, usually traceable to some error in compaction technique or calculation. A third test always checked within 5 per cent. with one of the first tests. Specific surface was calculated from the average value of S_v.

Tests and Results

The results of the size-surface measurements on quartz powders are given in Tables 19 and 20.

Table 19. - Size-Surface Measurements on Quartz Ottawa Sand, 20 minute grind

Sample	Fraction	Product	Wt. %	S	Surface (Sq. cm.)	% of total surface	De microns
Q 1	1	Q 9	29.7	780	232	14.1	24.0
	2	Q 10	13.6	1170	159	9.7	19.4
	3	Q 11	14.6	1660	243	14.8	13.6
	4	Q 12	5.9	2240	122	7.4	10.1
	5	Q 17	9.5	2980	282	17.2	7.6
	6	Q 18	11.0	5500	605	36.8	4.1
	Combined		84.5		1641		13.9
Q II	1	Q 13	30.4	715	217	12.6	31.8
	2	Q 14	14.2	1180	166	9.6	19.2
	3	Q 15	15.0	1660	250	14.5	13.6
	4	Q 16	6.2	2240	128	7.4	10.1
	5	Q 20	10.6	3190	336	19.5	7.1
	6	Q 21	11.4	5500	625	36.3	4.1
	Combined		87.8		1723		13.1
Q III	1	Q 23	28.5	740	211	12.6	30.5
	2	Q 24	14.3	1170	168	10.1	19.4
	3	Q 25	15.1	1620	245	14.7	13.9
	4	Q 26	12.7	2500	318	19.1	9.1
	5	Q 28	4.1	3500	143	8.1	6.5
	6	Q 29	10.5	5500	580	34.8	4.1
	Combined		85.3		1665		12.6
	Fraction		Wt. %		% Cum.	S	De
Average of above	1		29.5		29.5	745	30.4
	2		14.0		43.5	1175	19.3
	3		14.9		58.4	1645	13.7
	4	From QI	(6.0		64.4	2240	10.1
	5	and QII	(10.0		74.4	3080	7.4
	6		11.0		85.4	5500	4.1
Q VI	Unsized + Fraction 6		86.0			1690	13.4

Table 20. - Size Surface Measurements on Quartz Ottawa Sand. 10 minute grind

Sample Q VII					
Fraction	Product	Wt. %	S	De microns	
1	Q 32	52.5	680	33.2	
2	33	11.0	1320	17.1	
3	34	10.2	1770	12.8	(some sample lost in charging)
4	35	7.2	2600	8.7	
5	42	4.0	3600	6.3	
6	43	5.9	6000	3.8	
	Combined	90.8			

Sample Q VIII							
Fraction	Product	Wt. %	% Cum.	S	Surface sq. cm.	% of total surface	De microns
1	Q 37	55.3	55.3	680	378	26.4	33.2
2	38	11.2	66.5	1260	141	9.8	17.9
3	39	10.7	77.2	1640	176	12.3	13.8
4	40	7.8	85.0	2480	193	13.5	9.1
5	44	5.0	90.0	3650	183	12.8	6.2
6	45	6.0	96.0	6000	360	26.2	3.8
	Combined				1431		
+ Fraction 6	Q IX	91.5		1470			15.4
+ Fraction 6	Q X	91.7		1460			15.5

Q III is a sample of -200 mesh clean quartz from the 20 minute grind Ottawa Sand, elutriated by the modified procedure.

Q VII and VIII are 50 gram samples of the 10 minute grind Ottawa Sand product, elutriated by the modified procedure.

From the size distribution data and the specific surface of the fractions, the specific surface of all the material in each powder coarser than Fraction 7 was computed as shown. The distribution of the surface within this size range was also calculated for each sample.

The mean equivalent surface diameter, De for each fraction, and for each sample, is computed by the formula:

$$De = \frac{60,000}{d_1\ S}$$

where S is the specific surface

and d_1 is the density of the material = 2.65 gm. per cm.^{3}

Discussion of Results

Quartz samples I and II were run under identical conditions so an evaluation can be made of the reproducibility of the specific surface of the similar fractions from Table 19. The greatest difference is in Fraction 1, as Q 9 and Q 13 differ by 8.5 per cent. Since the probable error in surface determination is 7.3 per cent., this gives an error of only 1.2 per cent. directly attributable to reproducibility of the elutriation. The size distributions of the two samples agree very closely, as do the surface distributions.

Similar comparison with Q III is only possible for Fractions 1, 2,3, and 6, since Fractions 4 and 5 were obtained by the modified procedure using Tube 5. Again the agreement is good.

The specific surface as calculated for the combined Fractions 1 to 6, for Q I, Q II, and Q III, should be the same. Also this computed specific surface should check with the measured specific surface of Q VI. These specific surfaces all agree within 3 per cent., which is well within the probable error of 7.3 per cent.

Quartz samples Q VII and Q VIII, Table 20, also show good agreement in the specific surface of similar fractions. Comparison of the size distribution is only approximate, since some sample was lost in charging Q VII due to a connection failure.

The calculated specific surface for Q VIII differs from the average measured surface of Q IX and Q X by only 2.4 per cent.

A comparison of the specific surfaces of Q III and Q VIII, both elutriated by the same procedure, shows the effect of the fineness of grind on the reproducibility of the surface of equivalent fractions. Q III is from a 20 minute grind, -64 per cent. -200 mesh; Q VIII is from a 10 minute grind, 38.3 per cent. -200 mesh. Similar fractions differ in specific surface by 8.6 per cent. This difference is not any greater than that occurring between Q I and Q II on the same grind.

From the results of these tests, it seems that the time of grind from which the sample was taken, and the size distribution within the fraction, have very little effect on the specific surface of an elutriation fraction of the standard elutriation. The reproducibility of the standard elutriation is very good, the maximum error of 8.6 per cent. being mainly attributable to inaccuracy in surface determination.

Therefore standard surface values, determined for the standard elutriator fractions of a pure mineral, are sufficiently accurate to apply to a standard elutriation analysis without running individual surface determinations.

Also the size distribution of the product expressed in equivalent surface diameters can be determined. The size distributions for the -200 mesh products of the two grinds on Ottawa Sand are shown in Figure 29. The size distribution of a -200 mesh Cariboo Gold quartz product, obtained in later tests on ore minerals (Table 22), is also plotted for comparison.

Photomicrographs of Fractions 1 to 6 from Q II are shown in Figure 30. Surface measurement values for De are given for comparison with the linear dimensions. It may be seen that the linear dimensions

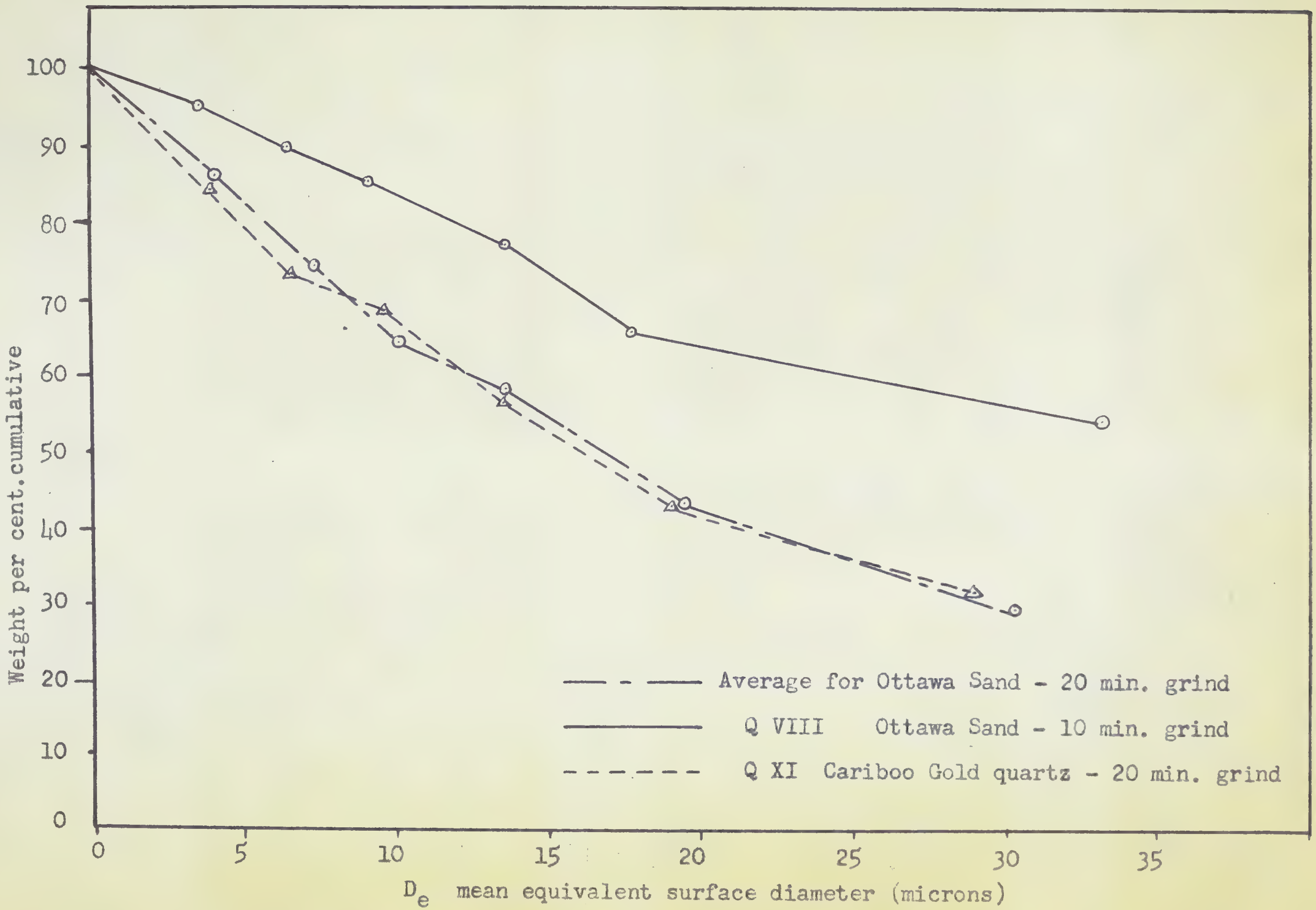
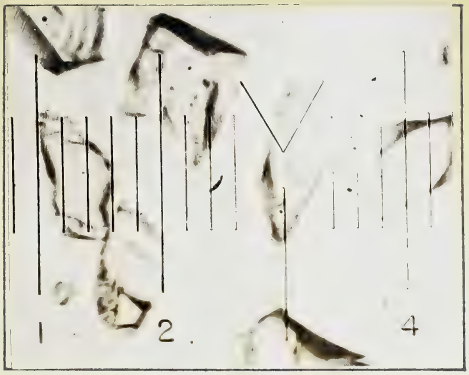
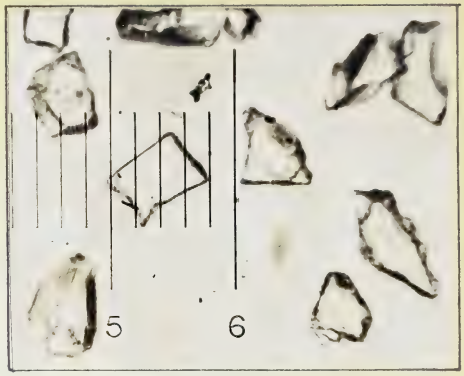


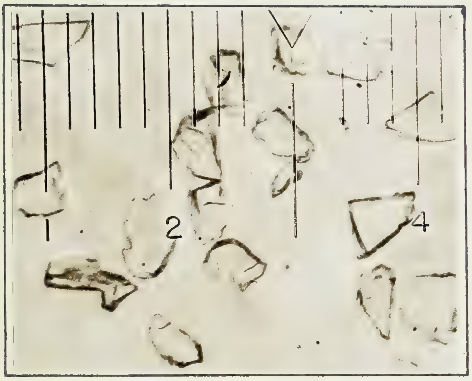
FIG. 29 - SIZE DISTRIBUTION OF QUARTZ SAMPLES



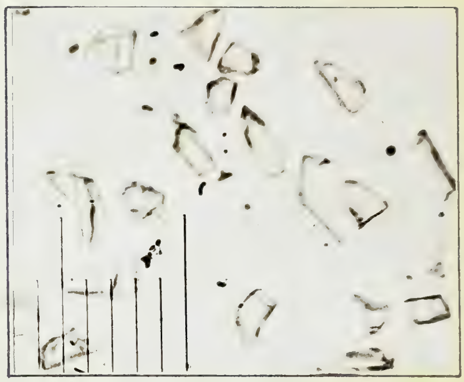
Fraction 1
D_e 31.8 microns



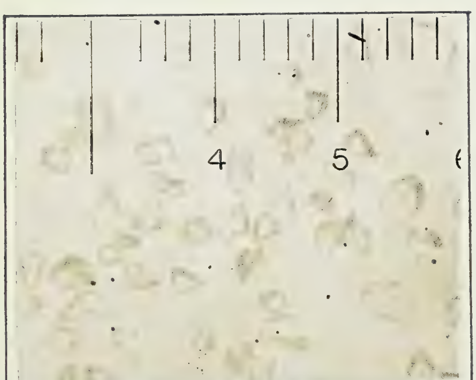
Fraction 2
D_e 19.2 microns



Fraction 3
D_e 13.6 microns



Fraction 4
D_e 10.1 microns



Fraction 5
D_e 7.1 microns

Fraction 6
D_e 4.1 microns

1 scale division 13.7 microns
D_e = equivalent surface diameter

FIG. 30 - PHOTOMICROGRAPHS OF QUARTZ FRACTIONS

differ considerably from the value of De due to the flaky shape of the particles.

STANDARD PROCEDURE FOR PURE MINERALS

It was apparent from the above experiments on quartz powders that a standard elutriation procedure could be calibrated, by use of the air permeability apparatus, to give a reasonably accurate determination of specific surface and size distribution. Therefore experiments were carried out to determine the calibration values for a number of pure minerals.

The elutriation apparatus as described above required modification to obtain a closer sizing of the coarser material with minerals of high specific gravity. Only one stage of elutriation was carried out on heavy minerals, since the finest fractions obtainable by the modified procedure were beyond the limits of the calibration curves. The procedure finally standardized involves standard elutriation of a -200 mesh product and surface determination on the standard fractions.

Standard Elutriation

The standard procedure is as shown in Table 21.

Table 21. - Standard Elutriation Procedure for Pure Minerals

Stage	Fraction	Flow Rate ml. per min.	Tube	Average Velocity (cm. per min.)	Nominal Size of Glass Spheres (microns)
I	A	175	A	16.8	+205.0
	B		B	19.2	+ 69.1
	1		1	7.67	+ 44.1
	2		2	4.56	+ 33.7
	3		4	2.23	+ 23.7
	4		5	0.99	+ 19.2
II	5	16	1	0.702	+ 13.2
	6		4	0.204	+ 7.2
	7		o'flo		- 7.2

The apparatus which was used is that shown in Figure 8. The supply system, flow regulator, charging apparatus, and flow measurement apparatus, are as used for glass spheres and quartz.

The elutriator tubes are six in number: straight tubes A and B, and separatory tubes 1, 2, 4 and 5. The dimensions and characteristics of these tubes are as given in Table 1. Flow in the straight tubes verges on turbulent, so the calculated limiting diameter for glass spheres is only approximate.

Stage II is not run on the heavy minerals, but is used for the lighter minerals. Weight of sample is varied depending on the specific gravity of the mineral, to avoid crowding in Tube A, and to give fractions large enough for surface determination.

Charging procedure must be carried out precisely as detailed in Steps (a) to (n) for Stage II of sphere elutriation. Otherwise rapid-settling heavy material will block the charger, or crowding will occur in Tubes A and B.

At the end of the run all tubes are drained, and the fractions are filtered, washed with alcohol, dried, and weighed.

Surface Determination

Surface determination is carried out as described for quartz powders, using the calibrated air permeability apparatus. If the size of sample is too small to use a standard bed depth, the depth must be measured after compaction with the vernier calipers, and 't' and 'w' calculated. The alignment chart, Figure 31, is used to calculate porosity.

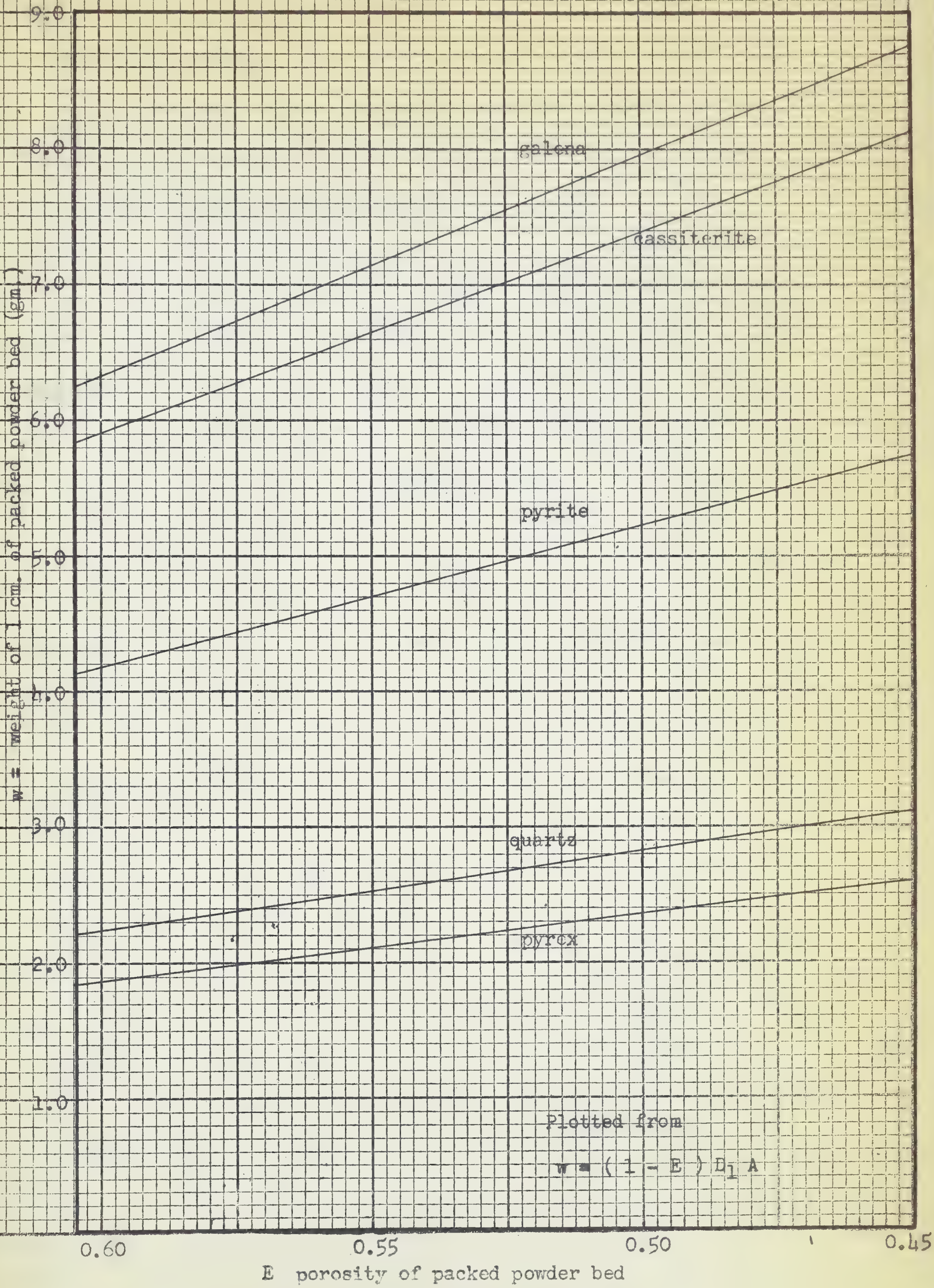


FIG. 31 - CHART FOR CALCULATION OF BED POROSITY

TESTS ON PURE MINERALS

Tests were carried out to determine the standard surface values of the standardized elutriation fractions on ground quartz, pyrex, pyrite, cassiterite, and galena.

Quartz

A hand picked sample of white rock quartz from Cariboo Gold Quartz ore was ground in the laboratory ball mill by the same procedure as was used for the Ottawa Sand. Time of grind was 20 minutes and the screen analysis of the product was:

Mesh	Weight per cent.
+ 65	1
+100	1
+150	4
+200	13
-200	81

The -200 mesh product was cleaned with hydrochloric acid, washed, and dried, as for the Ottawa Sand products.

A 50 gram sample, Q XI, was elutriated using the modified quartz elutriation procedure in Table 18. A 50 gram sample, Q XIII, was elutriated by the Standard Mineral Elutriation procedure in Table 21. Surface determinations were made on the quartz fractions. The results are given in Table 22.

The results check closely with the results of elutriation on Ottawa Sand quartz in Tables 19 and 20, with the difference that the coarse fraction is split in Q XIII, into Fractions B and 1.

The values for Q XIII may be taken as the calibration values for the standard elutriation fractions on this type of quartz.

Table 22. - Surface-Size of Standard Elutriation Fractions
Cariboo Gold Quartz

Sample	Fraction	Product	% Wt.	% Cum.	S	Surface Sq. cm.	De
Q XI	1	Q 46	32.1	32.1	780	250	29.0
	2	Q 47	11.7	43.8	1180	138	19.1
	3	Q 48	13.5	57.3	1680	227	13.4
	4	Q 49	12.0	69.3	2330	280	9.7
	5	Q 50	4.8	74.1	3350	161	6.7
	6	Q 51	11.9	86.0	5600	668	4.0
		Combined	86.0		1724	1724	13.1
Q XIII	A	nil	-	-	-	-	-
	B	Q 53	8.1	8.1	452	37	50.2
	1	Q 54	23.7	31.8	730	173	31.0
	2	Q 55	12.2	44.0	1130	138	20.0
	3	Q 56	14.2	58.2	1570	223	14.4
	4	Q 57	13.4	71.6	2340	313	9.7
	5	Q 59	5.2	76.8	3360	175	6.7
	6	Q 60	10.7	87.5	5600	600	4.1
		Combined	87.5		1659	1659	13.6

Ground Pyrex Glass

Two 40 gram samples of the ground pyrex powder used for sphere preparation, were elutriated by Stages I and II, with the results as given in Table 23. The overflow product from Stage I for each sample was very small, so both overflows were combined for Stage II to obtain Fractions G 16 and G 17.

Pyrite

A hand picked sample of crystalline pyrite from Cariboo Gold Quartz Ore was crushed in a steel mortar to -28 mesh, and ground to pass 100 mesh in a Braun Pulverizer. The screen analysis of the product was:

Mesh	Weight (grams)	Weight %
+ 100	0	0
+ 150	0.5	0.26
+ 200	10.0	5.24
− 200	180.7	94.5

Table 23. - Surface Size of Standard Elutriation Fractions

Ground Pyrex Glass

Sample	Fraction	Product	% Wt.	S	De
G II	A	nil	-	-	-
	B	G 6	12.2	500	54.0
	1	G 7	39.0	735	36.7
	2	G 8	16.3	1130	23.8
	3	G 9	16.1	1580	17.1
	4	G10	11.6	2320	11.6
G III	A	nil	-	-	-
	B	G11	11.3	560	47.5
	1	G12	36.6	760	35.0
	2	G13	17.0	1100	24.0
	3	G14	16.9	1500	17.9
	4	G15	12.3	2200	12.2
Average	B			530	50.7
	1			750	35.8
	2			1115	23.4
	3			1540	17.4
	4			2260	11.9
	5	G16	3.3	2940	9.2
	6	G17	2.2	4200	6.5

The specific gravity determined on crystalline material was 4.95.

A 50 gram pilot sample, P I, was elutriated to determine the operating technique. Tapping of the large tubes was necessary every 15 minutes.

Two 50 gram samples of the -200 mesh pyrite, P II and P III, were elutriated by Stage I only, with the results given in Table 24.

Table 24. - Surface-Size of Standard Elutriation Fractions

Pyrite

Sample	Fraction	Product	% Wt.	S	De
P II	A	P 12	1.2	-	-
	B	P 13	45.4	400	30.2
	1	P 14	16.9	730	16.6
	2	P 9	8.7	1100	11.0
	3	P 10	9.2	1530	7.9
	4	P 11	7.1	2400	5.0
P III	A	P 15	0.5	-	-
	B	P 16	43.4	346	35.0
	1	P 17	20.3	750	16.2
	2	P 18	8.9	1115	10.8
	3	P 19	9.0	1560	7.8
	4	P 20	6.6	2600	4.7
Average	A			-	-
	B			370	32.8
	1			740	16.4
	2			1110	10.9
	3			1550	7.8
	4			2500	4.9

Galena:

Hand picked, galena crystals, over one-eighth inch in diameter, were ground in a mortar to pass 65 mesh. The screen analysis of the product was:-

Mesh	Weight (grams)	Weight %
+ 100	93	23
+ 150	32	8
+ 200	52	13
- 200	227	56

The specific gravity, determined on crystals, was 7.50.

Two 80 gram samples of the 200 mesh galena, L I and L II, were elutriated, by Stage I only, to obtain the results in Table 25.

The specific surface of Fraction 4 was obtained by theoretical calculation.

Table 25. - Surface-size of Standard Elutriation Fractions

Galena

Sample	Fraction	Product	% Wt.	S	De
L 1	A	L 1	11.4	170	47
	B	L 2	43.5	250	32
	1	L 3	13.9	560	14.3
	2	L 4	6.5	790	10.1
	3	L 5	7.8	1140	7.0
	4	L 6	4.7	(part lost in charging)	
L 2	A	L 7	11.0	187	42.7
	B	L 8	44.8	252	31.6
	1	L 9	13.6	530	15.0
	2	L10	6.0	785	10.2
	3	L11	7.9	1250	6.4
	4	L12	7.7	2400	3.3
Average	A			179	44.5
	B			251	31.8
	1			545	14.6
	2			787	10.2
	3			1200	6.7
	4			2400	3.3

Cassiterite

Two 80 gram samples of a -200 mesh cassiterite table concentrate were elutriated by Stage I to obtain the results in Table 26. Microscopic analysis of the fractions showed Fractions A, B, and 1 were 99 per cent. cassiterite, the specific gravity being determined as 7.00 by specific gravity bottle. Fractions 2, 3, and 4 showed impurities of heavy sulfides and garnet so were discarded.

Table 26. - Surface-size of Standard Elutriation Fractions

Cassiterite

Sample	Fraction	Product	% Wt.	S	De
C I	A	C 1	7.1	218	39.4
	B	C 2	77.0	287	29.8
	1	C 3	14.4	690	12.4
	2	C 4	1.9		
	3	C 5	.50		
	4	C 6	.25		
C II	A	C 7	8.7	211	40.5
	B	C 8	72.0	284	30.0
	1	C 9	17.0	720	11.9
	2	C10	1.9		
	3	C11	.54		
	4	C12	.21		
Average	A			215	40.0
	B			286	29.9
	1			605	12.2

Discussion of Results

For each mineral the specific surface values, for the fractions obtained as products of the separatory tubes, check within the limits of 8.6 per cent. as determined for quartz. The difference between the fractions obtained in the straight tubes is as large as 13 per cent. (for pyrite). This is to be expected due to the turbulent conditions in the tubes and the greater error inherent in the surface determination with very coarse powders. However, the error in the coarse fractions is not serious, since only a small percentage of the total surface of the powder is involved.

The percentage weight retained by Tube A was small, even with the heavy minerals, indicating that a larger tube should be used if sizing of pyrite and quartz powders above the average equivalent surface

diameter of 40 microns is desired. Again only a small percentage of the total surface is involved.

The procedure for elutriation is very satisfactory for the purpose. The reproducibility is shown for quartz samples in Tables 19 and 20, and for pure minerals in Tables 22 to 26. The deviation in weight for any pair of similar fractions is in almost all cases less than 1.5 per cent.

The values of the equivalent spherical diameter, De, are computed from the results for each fraction. This provides a means for obtaining a size distribution for each mineral powder. However, the size is based on surface, not on linear dimensions.

The standard specific surface values for the elutriator fractions, for all the pure minerals tested, are summarized in Table 27. The specific surfaces of the equivalent spherical fractions are included.

Since specific surface involves the factor of specific gravity, the size of the mineral particles in corresponding fractions can be better compared on the basis of average equivalent surface diameter, D_e. Values of D_e are summarized in Table 28 and shown graphically in Figure 32.

An idea of the actual particle shapes and sizes can be gained from the photomicrographs of the mineral fractions in Figures 30 and 33. The shape of the quartz particles in Figure 30 appears to change as the size becomes smaller. In Figure 33 the cassiterite sample shown is Fraction 1. All other photomicrographs are of Fraction 2. The apparent difference in the size of the pyrex powder particles and the pyrex spheres is due to the flaky shape. The cubical tendency of the galena particles is apparent.

Table 27. - Specific Surface of Standard Elutriation Fraction of Pure Minerals

Standard Fraction	Pyrex Spheres	Pyrex Powder	Quartz	Pyrite	Galena	Cassiterite
A					129	215
B		530	452	370	251	286
1	540*	750	730	740	545	605
2	760	1115	1130	1110	787	
3	1000	1540	1570	1550	1200	
4	1290*	2260	2340	2500	2400	
5	1710*	2940	3360			
6	2660	4200	5600			

* not run under mineral standard conditions.

Table 28. - Equivalent Surface Diameters for Standard Elutriation Fractions of Pure Minerals

Standard Fraction	Average Elutria-tion Vel. cm./min.	De (microns)					
		Pyrex Spheres	Pyrex Powder	Quartz	Pyrite	Galena	Cassiterite
A	168					44.5	40.0
B	19.2	-	50.7	50.2	32.8	31.8	29.9
1	7.67	50.0	35.8	31.0	16.4	14.6	12.2
2	4.56	35.4	23.4	20.0	10.9	10.2	
3	2.23	26.9	17.4	14.4	7.8	6.7	
4	0.990	18.0** 20.9*	11.9	9.7	4.9	3.3	
5	0.702	15.7	9.2	6.7			
6	0.204	10.3	6.5	4.1			

** From Fig. 32 for 0.99 cm. per min.
* elutriation velocity 1.43 cm. per min.

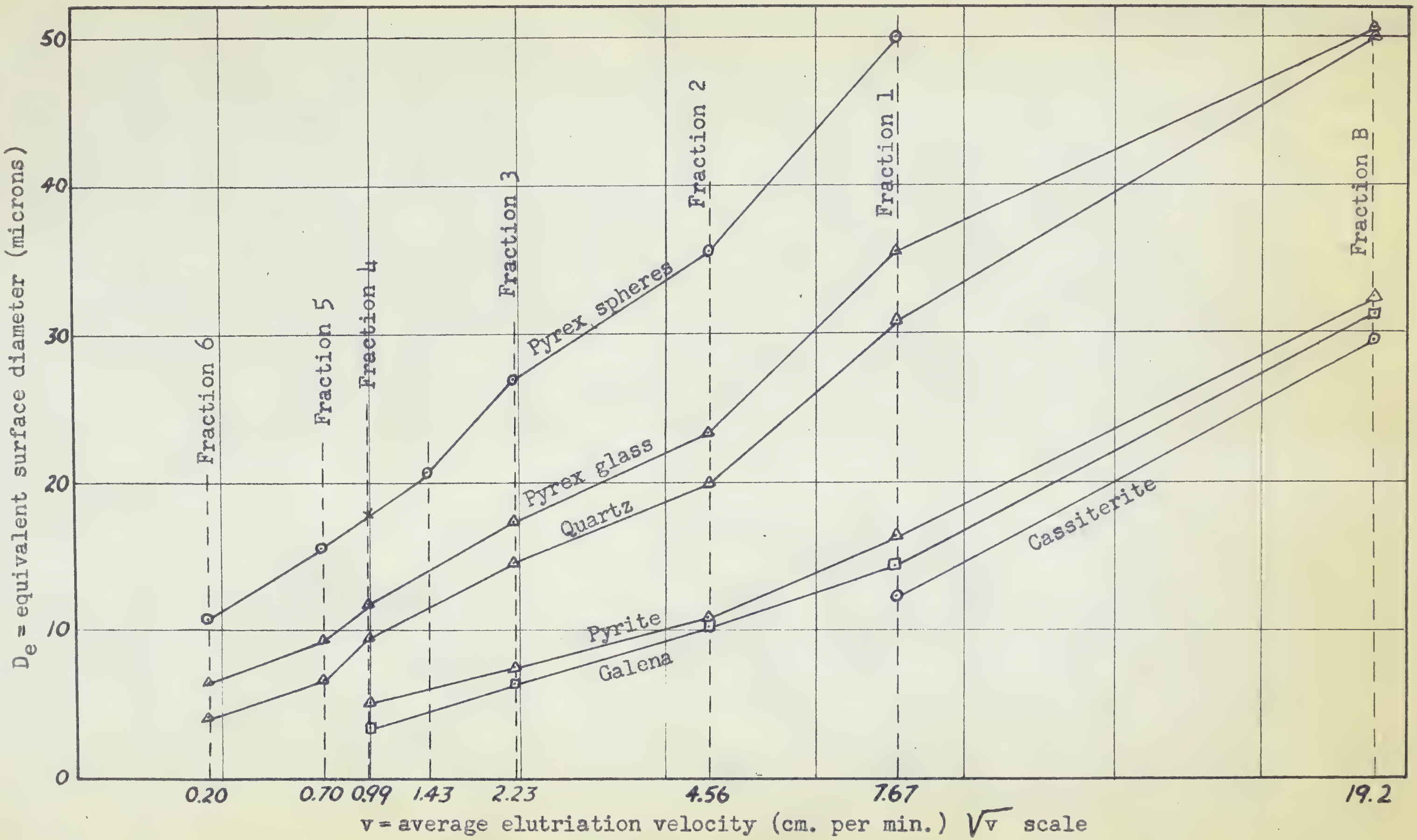
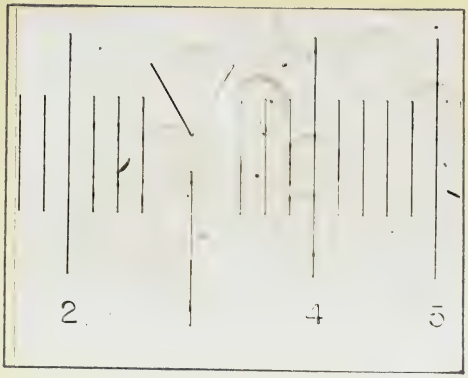
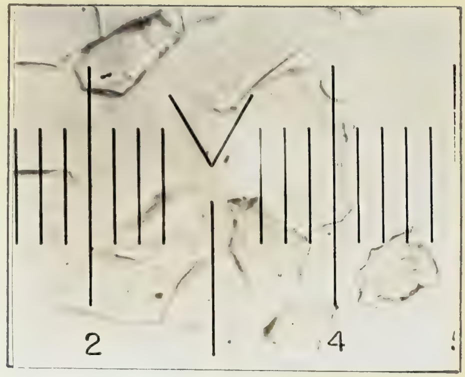


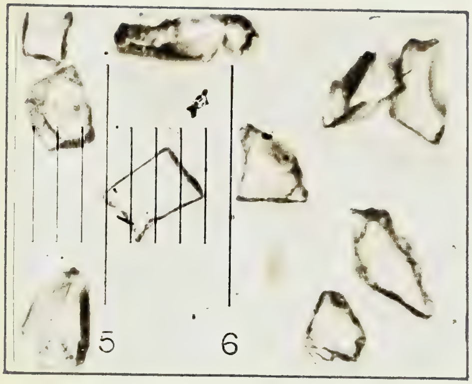
FIG. 32 - EQUIVALENT SURFACE DIAMETER FOR STANDARD ELUTRIATOR FRACTIONS



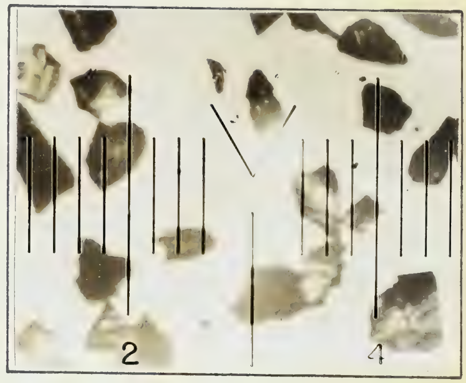
Pyrex Spheres
Fraction 2 D_s 35.9



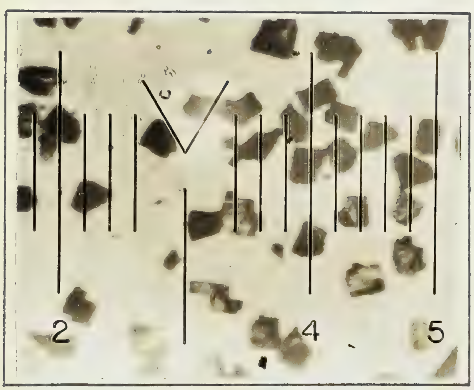
Ground Pyrex
Fraction 2 D_e 23.4



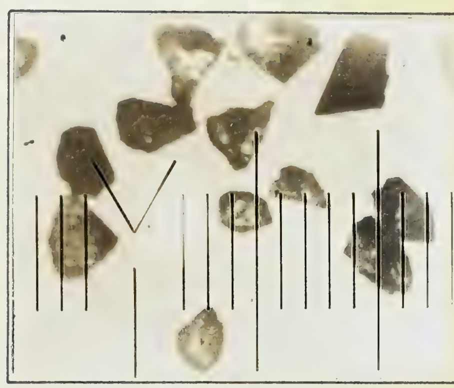
Quartz
Fraction 2 D_e 19.2



Pyrite
Fraction 2 D_e 10.9



Galena
Fraction 2 D_e 10.2



Cassiterite
Fraction 1 D_e 12.2

1 scale division 13.7 microns
De = equivalent surface diameter (microns)

FIG. 33 - PHOTOMICROGRAPHS OF MINERAL FRACTIONS

Exception may be taken to the method of grinding for pyrite and galena. The method used was dictated by the small amount of pure crystalline material available. The shape of the particles would probably vary from that in products prepared by ball mill grinding. However there is no reason to expect that the accuracy of the standard procedure would vary for minerals ground by any other method although actual calibration values would vary.

APPLICATION TO A HETEROGENEOUS POWDER

As a preliminary to application of the surface calibrated elutriation procedure to heterogeneous ores, tests were carried out on synthetic samples made up from pure quartz and pyrite fractions.

Surface Measurements on Heterogeneous Samples

The first investigation was to confirm the applicability of the calibrated air-permeability surface determination to heterogenous mixtures of quartz and pyrite.

Four binary mixtures were made up from quartz and pyrite fractions of known specific surface. Each mixture contained 2.000 grams of the equivalent standard fraction of each pure mineral. The composition of each composite sample is given in Table 29.

The specific gravity of the 50 per cent. pyrite mixtures is 3.44, as computed at Appendix IV.

A surface determination was made on each mixture. The agreement between the calculated and measured specific surface is shown in Table 29. The maximum difference of 5.1 per cent. is well within the probable error of 7.3 per cent. discussed previously.

Therefore the calibrated air-permeability apparatus is as accurate on binary mixtures as on pure minerals.

Table 29. - Surface Determinations on Pyrite - Quartz Composites

Composite	Standard Fraction	Components			Composite Specific Surface		
		Product	%	S	Calculated	Measured	Difference %
PQ.A	1	Q 54	50	730			
		P 17	50	750	740	780	5.1
PQ.B	2	Q 55	50	1130			
		P 18	50	1115	1122	1110	1.1
PQ.C	3	Q 56	50	1570			
		P 18	50	1560	1565	1620	3.5
PQ.D	4	Q 57	50	2340			
		P 20	50	2600	2470	2400	2.9

Size-Surface Measurements on Heterogeneous Mixtures

The second investigation was to determine the accuracy of the elutriation- surface determination procedure on a pyrite-quartz composite sample.

The head sample was made up of the four pyrite quartz mixtures whose surface was measured in the previous investigation. Its composition and calculated specific surface are given in Table 30.

Elutriation was carried out by the standard procedure with the results as shown in Table 30. The specific surface of Fractions 1, 2, 3, and 4 was measured by the air permeability apparatus. The specific gravity of each fraction was assumed to be 3.44.

The specific surface of the composite was recalculated using the elutriation results. A small fraction of material was found to overlap the range of head sample fractions at both ends. Standard surface values for Fractions B and 5 were assigned to this material from Table 27. The recalculeted specific surface checked within 3 per cent. with the calculated specific surface for the head sample.

Table 30. - Elutriation Test on a Pyrite-Quartz Composite

Head Samples						Elutriation Products				
Fraction	Components	Weight grams	Weight %	S Calculated	Calculated surface in 1 gm.	Product	Weight grams	Weight %	S Measured	Calculated surface in 1 gram
B						PQ 1	0.28	1.8	-	8*
1	PQ A	3.95	25.2	740	187	PQ 2	4.40	28.4	825	234
2	PQ B	3.93	25.1	1122	279	PQ 3	3.61	23.3	1120	261
3	PQ C	3.90	24.9	1565	390	PQ 4	3.79	24.4	1550	378
4	PQ D	3.90	24.9	2470	613	PQ 5	3.00	19.6	2330	457
5						PQ 6	0.40	2.6	-	87*
						loss	0.20	1.3		
Total		15.68		1469	1469	Total	15.68			1425

* Calculated from standard surface value - Table 27.

Although the weight distribution in the fine and coarse elutriation products varied about 5 per cent. from the weight distribution of the head sample, the procedure is sufficiently reproducible for any practical use.

Discussion

It would seem, from the preliminary work on heterogeneous mixtures, that the surface determination procedure, using the air-permeability apparatus or the calibrated elutriation surface determination procedure, can be applied successfully to simple heterogeneous ores.

Additional procedure would be necessary for determining the mineral composition of the elutriator products, so that specific density could be calculated and the standard surface values applied correctly. In most cases a chemical analysis for the key metal constituents would provide data from which the mineral composition could be calculated. This would be possible if the minerals were of definite composition such as the common sulfides, but would not be practicable if the minerals were mainly oxidation products or other minerals of variable composition. Direct determination of specific gravity of each product by physical methods would enable the specific surface to be measured by the air-permeability apparatus.

SUMMARY

The air-permeability apparatus may be used on any dry mineral powder to determine the specific surface directly. The result is not affected significantly by the size distribution of the powder or the number of mineral components.

For size-surface analysis the elutriation procedure, with the specific surface of the elutriator fractions previously established by air-permeability, is applicable to all homogeneous mineral powders.

Application of the air-permeability method to heterogeneous mineral powders is dependent upon the accurate determination of specific gravity. In order to apply the elutriation procedure to these powders the mineral composition must be determined for each fraction.

Although the results on two different grinds of quartz (38.3 and 64.1 per cent. minus 200 mesh) show no significant difference in the specific surface of equivalent standard fractions, it is possible that different grinding machines, charges, or critical speeds, may produce materials with a great enough difference in shape to cause a difference in the standard surface values. Therefore the values in Tables 27 and 28 may be considered as standard only for the conditions given. For any other grinding procedure, standard values should be determined by use of the air-permeability apparatus on each elutriator fraction. These standard values should then be applicable to the elutriator fractions from any subsequent mill product.

CONCLUSIONS

The U-tube type air-permeability apparatus may be used with reasonable accuracy to determine the specific surface of mineral dressing products. If calibrated against powders of known specific surface the porosity range is limited only by the requirements of uniform compaction. However the theoretical calculation of specific surface by the Modified Rigden Equation is in close agreement with calibration results for porosities between 0.50 and 0.60, which are

readily obtained with all angular materials tested. Therefore calibration curves may be set up from theoretical calculations, corrected for filter paper error, for routine specific surface determination.

The air-permeability apparatus may be used on any dry material to determine the specific surface directly. The result is not affected significantly by the size-distribution of the powder or the number of mineral components. The apparatus is simple to construct, portable and easily operated. The only special technique required is that necessary for preparation of a uniformly compacted powder bed in the permeability cell. A complete test can be made in less than 10 minutes if calibration charts are available.

The air permeability apparatus may also be used to determine the distribution of surface within a mineral powder. The powder must first be elutriated to obtain a set of fractions whose weight distribution is known. If then the surface of each fraction is measured by the air-permeability apparatus, a surface distribution is obtained, which can be converted if desired into an equivalent surface size distribution.

However if the elutriation procedure is standardized, the specific surface and equivalent surface diameter, as determined for a pure mineral for any elutriator fraction, can be taken as standard. Once this calibration is made, only elutriation and application of the standard values are required to obtain a surface or size analysis.

Although the method of elutriation used in the investigation was very satisfactory, there is no reason why an equally reproducible elutriation method cannot be used. Air elutriators, such as the

Haultain infrasizer, may even be preferable since they yield dry products.

The standard surface and spherical-diameter values must be determined on the pure minerals for a given ore. Since at the present time the effect of grinding procedure on the shape of particles is not determinable, the standard surface tests should be made on the products of each grinding method. Once determined for a given method they will be usable regardless of operational changes. In dealing with heterogeneous ores the pure minerals could be recovered from the mill product by selective flotation or heavy-media separation.

Although the size-surface analysis procedure must be further developed for use on heterogeneous ores, its application to two or three component ores should be simple. It is possible that it cannot be fully applied successfully to highly complex ores containing minerals of variable composition. However the method can be used in conjunction with chemical analysis to determine the surface of certain key minerals. Regardless of the other minerals present, the standard size-surface values for a given ore mineral will hold for the standard elutriator fractions.

The direct surface determination, and the size surface analysis applications of the air permeability method to the evaluation of surface in ore dressing products will provide sound data upon which the operation and control of mineral dressing operations can be based. Surface efficiency relationships should be based, not on estimation and guess work, but on measurement.

RECOMMENDATIONS

Although the method described in the above investigations is applicable in the present form, improvements are indicated.

The air-permeability apparatus would be improved by a smaller cell, say one centimeter in diameter, to permit of the use of a smaller sample. This would also permit of a general reduction in the size of the apparatus, particularly in the capacity of the U-tube. A design similar to the Blaine Apparatus would seem to be indicated if provision were made for increasing the length of the powder bed for coarse samples. Uniformity of compaction would require re-investigation with the smaller cell. A precision timer, accurate to one-fifth second, would be desirable.

In the elutriation procedure, better sizing in the coarse range could be obtained by a better choice of straight tubes. A means of regulating the temperature to within one degree would eliminate the appreciable error introduced by variable temperature. There is no apparent reason why the size range could not be extended to finer material by use of lower velocities in the larger tubes, although the utility of the results is doubtful.

The surface range of the curves as calibrated by the glass spheres is inadequate for the heavier minerals, and although the theoretical calculation is acceptable, its validity should be checked for higher surface values. Preparation of the necessary minus 7 micron glass spheres is possible by the method described, but the accurate microscopic evaluation of their specific surface could only

be carried out by use of monochromatic light and a better condenser system, or by the electron microscope.

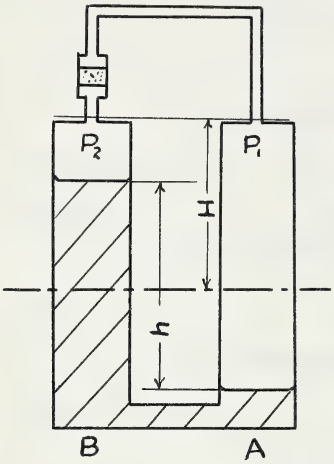
It is apparent that investigation of the effect of the grinding procedure on particle surface should be investigated, so that the applicability of the method of size-surface analysis to all mill products can be determined.

The applicability of the method to mineral products suggests many problems for investigation, the number of which is indicated by the numerous application of particle surface knowledge to the problems of ore dressing.

APPENDICES

Appendix I

Derivation of the Equation for the Rigden Apparatus



Considering the U-tube apparatus in the diagram, where P_1 and P_2 are the pressures above the oil columns in A and B and h is the difference in oil level, if d_2 is the oil density and d_1 is the air density,

then $P_1 - P_2 = hg\,(d_2 - d_1)$.

If G is substituted for $g(d_2 - d_1)$

then $P_1 - P_2 = hG$. (1)

Since the mass of air in both tubes is constant for any position of the oil level

$$(H + \frac{h}{2})\, a\,\sigma\, P_1 + (H - \frac{h}{2})\, a\,\sigma\, P_2 = 2\, H\, a\,\sigma\, P_o$$

where σ = density of air at unit pressure,

and a = cross section area of the tubes.

or $H\,(P_1 + P_2) + \frac{h}{2}\,(P_1 - P_2) = 2\,H\,P_o$. (2)

Substituting from (1)

$$P_1 + P_2 = 2\,P_o - \frac{h}{2H}\,(h\,G) \qquad (3)$$

Adding equations (1) and (3)

$$P_1 = P_o + (1 - \frac{h}{2H})\,\frac{hG}{2} \qquad (4)$$

If V_1 = volume of air passing from A into the bed per second, then the mass of air passing into the bed = $V_1 P_1 \sigma$. This equals the rate of change of the air mass in A per second = $-\frac{d}{dt}(H + \frac{h}{2})\, a\, P_1 \sigma$

Then

$$V_1 P_1 = \frac{d}{dt}(H + \frac{h}{2})\, a\, P_1 \qquad (5)$$

For an incompressible fluid, the Kozeny Equation (see page 39) gives $V = (P_1 - P_2) K_1$ (6)

where $K_1 = \frac{A\,E^3}{k\,(1 - E)^2\, U\, S^2\, d_1{}^2\, L}$, the permeability constant for the bed.

For a compressible fluid assuming $\frac{dP}{dx}$ constant along the axis of the bed

$$P_1 V_1 = P_2 V_2 = \frac{P_1 + P_2}{2}(P_1 - P_2) K_1 \qquad (7)$$

where P_1 and P_2 are entrance and exit pressures and V_1 and V_2 are volumes of gas entering and leaving the bed per second.

Combining (7) and (5):

$$\frac{P_1 + P_2}{2}(P_1 - P_2) K_1 = \frac{d}{dt}(H + \frac{h}{2})\, a\, P_1$$

Substituting for P_1 and P_2 from (1), (3) and (4)

$$\frac{2\,P_o - \frac{h}{2H}(h\,G)}{2} .\; h\,G\, .\, K_1 = \frac{d}{dt}(H + \frac{h}{2})\, a \left\{P_o + \frac{hG}{2}\left(1 - \frac{h}{2H}\right)\right\}$$

simplifying and integrating between t_1 and t_2 and h_1 and h_2

$$2\,\frac{KG}{a}\,\Big[t\Big]_1^2 = -\Big[\ln h\Big]_{h_1}^{h_2} + \frac{GH}{P_o}\left[-\ln h + \left(\frac{h}{2H}\right)^2\right]_{h_1}^{h_2} \qquad (8)$$

The final term in (8) may be discarded if h_1 and h_2 are kept small as compared to H.

Then $\frac{2\,K_1 G\,t}{a} = -\,(\ln h)_{h_1}^{h_2}$ (9)

Since σ is small compared to d, $G \triangleq hgd_2$

Substituting for G and K in (9)

$$\frac{2\,A\,\varepsilon^3\,ghd_2t}{k(1-\varepsilon)^2\,U\,S^2\,d_1^2\,L\,a} = -\,(\ln\ h)_{h_1}^{h_2}$$

which may be rearranged into the form

$$S^2 = \frac{2\,g\,d_2}{a\,\ln\frac{h_1}{h_2}} \cdot \frac{AT}{k\,U\,L\,d_1^2} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2}$$ as used with the

apparatus.

Appendix II

<u>Sample Calculation for $\sqrt{t}$ by the Modified Rigden Equation</u>

From theory $\sqrt{t} = \dfrac{Sd_1}{\sqrt{K}\ \sqrt{f(E)}\ \ \sqrt{C}}$ (pg. 139)

where K = constant for the apparatus with bed depth 1 cm.

$$= \frac{2\ gd_2}{a\ \ln \frac{h_1}{h_2}} \cdot \frac{A}{kU}$$

$$C = 1 + \frac{Sd_1(1 - E)\ Z\lambda}{E}$$

and $f(E) = \dfrac{E^3}{(1 - E)^2}$

For the apparatus at 25°C.

$g = 980$

d_2 = density of U-tube fluid = 1.108 gm. per ml.

a = cross section of U-tube = 7.22 cm.^{2}

h_1 = height of top timing mark = 13 cm.

h_2 = height of bottom timing mark = 3 cm.

A = cross section of cell = 2.11 cm^2

U = viscosity of air = 0.000182 poises

then $K = 47.50 \times 10^4$

and $\sqrt{K} = 687.$

For air at 25°C.

λ = mean free path of molecules = 9.6×10^{-6} cm.

Z = slip factor = 3.3 (experimental value used by Carman[52])

Assuming E = fractional porosity of bed = 0.60

and S = specific surface of pyrex powder = 2000 cm.^{2} per gm.

where d_1 = density of pyrex = 2.23 gm. per cm.^{3}

by calculation

$C = 1.0933$

and $\sqrt{C} = 1.045$

$$f(E) = \frac{E^3}{(1 - E)^2} = 1.35$$

$$\sqrt{f(E)} = 1.16$$

Then $$\sqrt{t} = \frac{Sd_1}{\sqrt{K}\ \sqrt{f(E)}\ \sqrt{C}}$$

$$= \frac{2000 \times 2.23}{687 \times 1.16 \times 1.045}$$

$$= 5.36 \text{ sec.}^{1/2}$$

Appendix III

Calculation of Specific Surface from Permeability Test

From theory $S = \dfrac{\sqrt{K}\sqrt{t}\;\sqrt{f(E)}\;\sqrt{C}}{d_1}$ (1)

where terms are as in Appendix II.

Since $C = 1 + Sd_1 \dfrac{1 - E}{E} Z \lambda$ (2)

calculation of S from (1) involves solution of a binomial equation. To avoid this solution a preliminary estimate of S will usually give a close approximation for $\sqrt{C}$.

Assuming $E = .555$

and $\sqrt{t} = 19.0$ from a permeability test where S is estimated at 6000 sq. cm. per gm. for a quartz powder ($d_1 = 2.65$).

then $C = 1 + 0.412 = 1.412$

$\sqrt{C} = 1.19$

$\sqrt{f}\,(E) = 0.935$

$S = \dfrac{687 \times 19.0 \times 0.935 \times 1.19}{2.65}$ (Using constants from Appendix II)

$= 5500$ sq. cm. per gm.

Recalculation of C using this value of S will eliminate the error due to estimation.

Appendix IV

Calculation of Specific Gravity of Binary Mixtures

If in one gram of a binary mixture there are x grams of material A, specific gravity d_1, and (1-x) grams of material B, specific gravity d_2, and the specific gravity of the mixture is d, then volume of component A in one gram is $\frac{x}{d_1}$,

and volume of component B is $\frac{1 - x}{d_2}$,

while volume of one gram of mixture is $\frac{1}{d}$.

Therefore $$\frac{x}{d_1} + \frac{1-x}{d_2} = \frac{1}{d}$$

$$\text{or } d = \frac{d_1\ d_2}{xd_2 + (1-x)d_1}$$

For a mixture 50 per cent. pyrite (sp. gr. 4.95) and 50 per cent. quartz (sp. gr. 2.65)

$$d = \frac{4.95 \times 2.65}{.5 \times 2.65 + 5 \times 4.95}$$

$$= 3.44$$

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